FTIR for Food & Flavors

More than 60 years of identifying and confirming both target and unknown molecules

1947
First commercial recording UV-Vis, the Cary 11 UV-Vis

1954
Release of the Cary 14 UV-Vis-NIR

1969
First rapid-scanning Fourier transform infrared spectrometer, the FTS-14

1979
First use of a mercury cadmium telluride (MCT) detector in a FTIR

1982
First FTIR microscope, the UMA 100

1989
Release of the acclaimed Cary 1 and 3 UV-Vis

1999
First 256 x 256 MCT focal plane array for analytical spectroscopy

2000
First ATR chemical imaging system

2007
Smallest, most rugged commercially available interferometer introduced

2007
TumblIR sample accessory introduced – a revolution in FTIR liquid sampling

2008
First handheld FTIR, the ExoScan

2011
The Cary 630 FTIR raises the bar for routine analysis of solids, liquids, and gases

2014: Next-generation, 4300 Handheld FTIR introduced

Agilent’s FTIR Family

Mobile	Analyzers	Entry	High
Routine	Routine	Routine	Research
Materials Chemicals Art Conservation	Petrochem Biodiesel	Chemicals Pharma Academia	Materials Polymers Academia Bio-Research

Agilent has the widest range of FTIR instruments – from Mobile to Routine to Research to Microscopy

RELIABLE: From the Lab to the Loading Dock

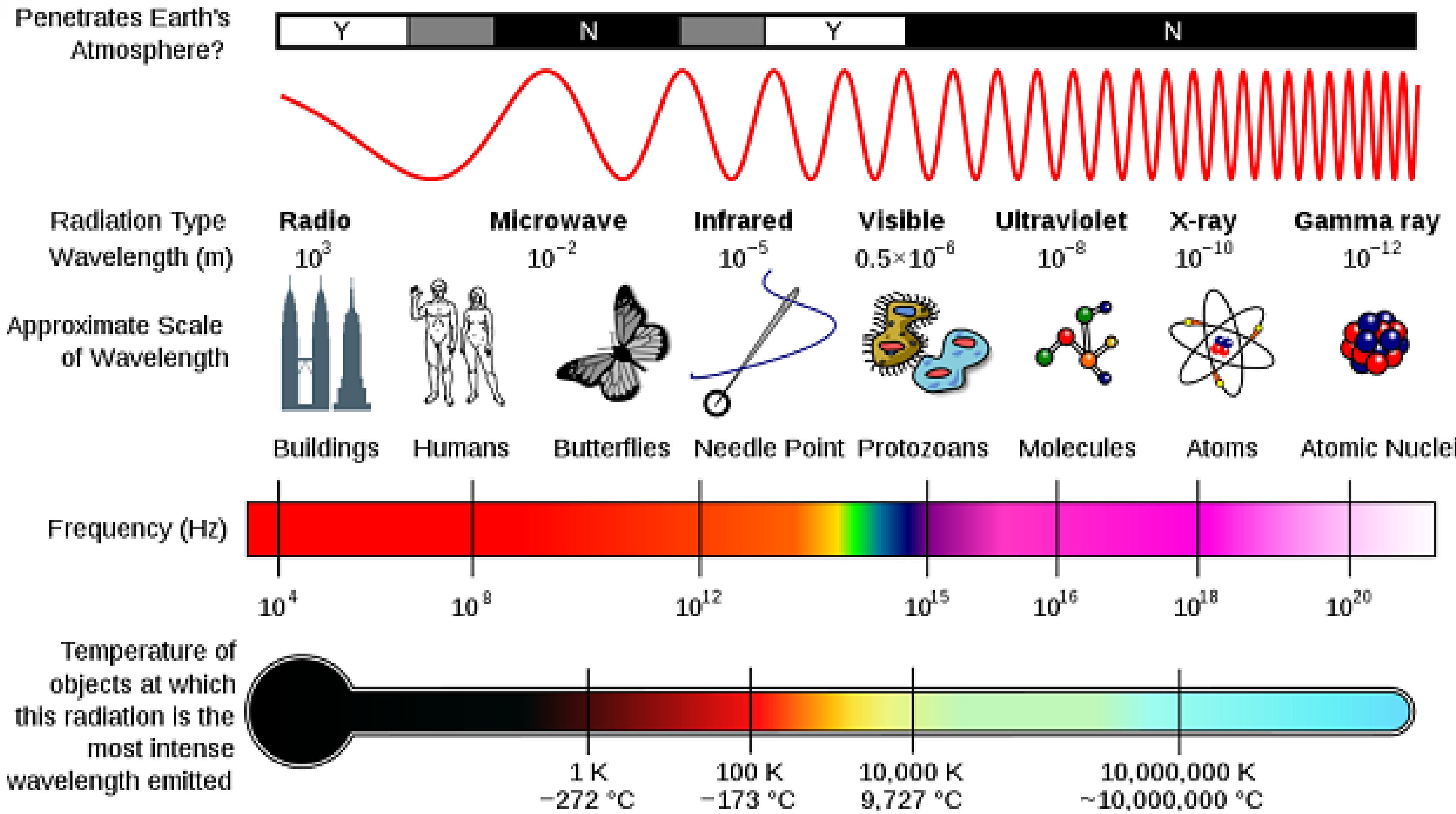
Handheld

Portable

Analyzers

Laboratory

The Electromagnetic Spectrum

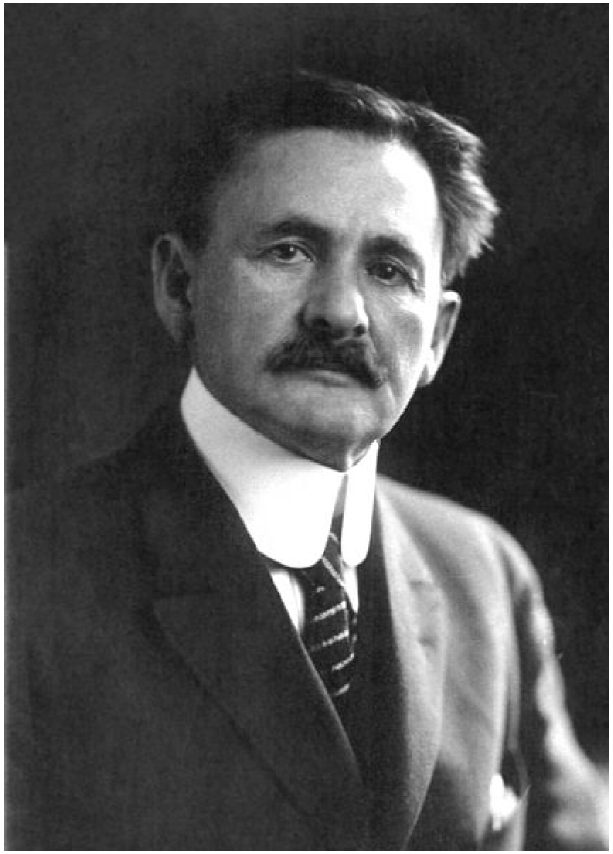


What does the F stand for in FTIR

Jean Baptiste Joseph Fourier
(21 March 1768 – 16 May 1830)

Was a French mathematician and physicist born in Auxerre and best known for initiating the investigation of Fourier series and their applications to problems of heat transfer and vibrations. The Fourier transform and Fourier's Law are also named in his honor. Fourier is also generally credited with the discovery of the greenhouse effect.

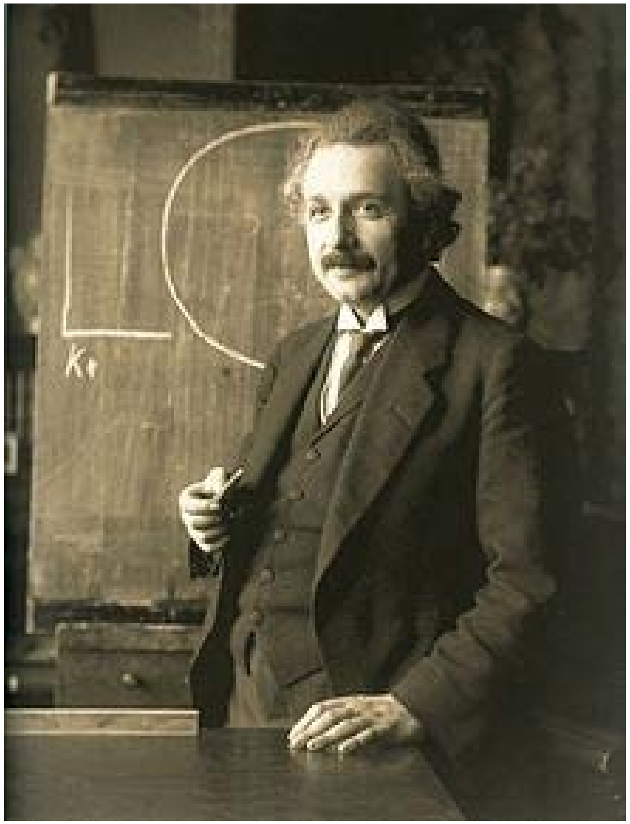
The Speed of light



Albert Abraham Michelson,
(December 19, 1852 – May 9, 1931)

Michelson was an American physicist known for his work on the measurement of the speed of light and especially for the Michelson–Morley experiment. In 1907 he received the Nobel Prize in Physics. He became the first American to receive the Nobel Prize in sciences. From 1920 and into 1921 Michelson and Francis G. Pease became the first individuals to measure the diameter of a star other than the Sun. They used an astronomical interferometer at the Mount Wilson Observatory to measure the diameter of the super-giant star Betelgeuse.

Einstein – Special relativity



Special relativity was originally proposed in 1905 by Albert Einstein in the paper "On the Electrodynamics of Moving Bodies". Galileo Galilei had already postulated that there is no absolute and well-defined state of rest (no privileged reference frames), a principle now called Galileo's principle of relativity. Einstein extended this principle so that it accounted for the constant speed of light,[5] a phenomenon that had been recently observed in the Michelson–Morley experiment.

Agilent is the premier measurement company for the food industry

We have many applications for food safety and quality across all product lines

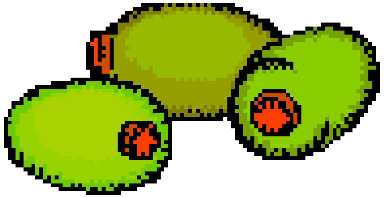
- Mycotoxins (LC-QQQ-MS)
- Pesticide screening (GC-MS)
- Fish ID (BioAnalyzer)
- Metals in Food (ICP-MS)
- Pathogen Detection (LC-MS, GC-MS)
- PCB (GC-QQQ)
- PAH (LC, GC, MS)
- Antibiotics in Food (LC, GC, MS)
- Adulteration (LC, GC, FTIR)
- Coffee, Tea, Sugar, Flour, Dairy testing (FTIR)
- Many, many more published, plus many on the way!!

Opportunity

The demand for applications is growing significantly faster in the food testing industry than in any other segment

Verification of Food Oils composition

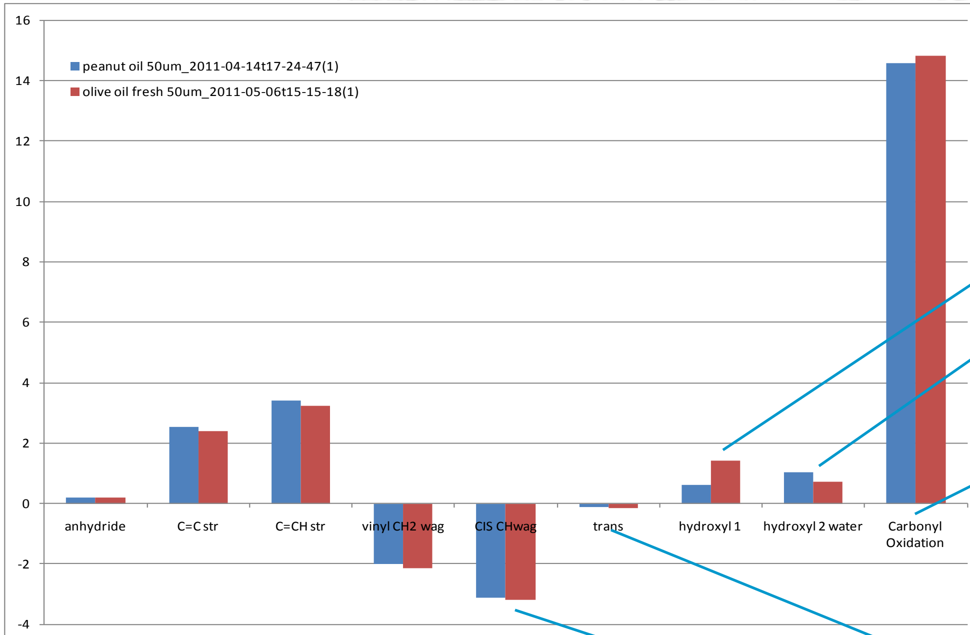
- Certain food oils sell at a high price but are easily diluted with less expensive but similar oils that affect the taste or flavor
- Verification of oil type important both economically and for customer satisfaction
- Although similar in basic chemistry, ratios of specific functional groups differ between types of oil
  - Hydroxyl, carbonyl, saturated and unsaturated aliphatic groups
- Agilent DialPath technology can be used to verify oils quickly
  - Quantitative method
  - Band Ratios used for identification

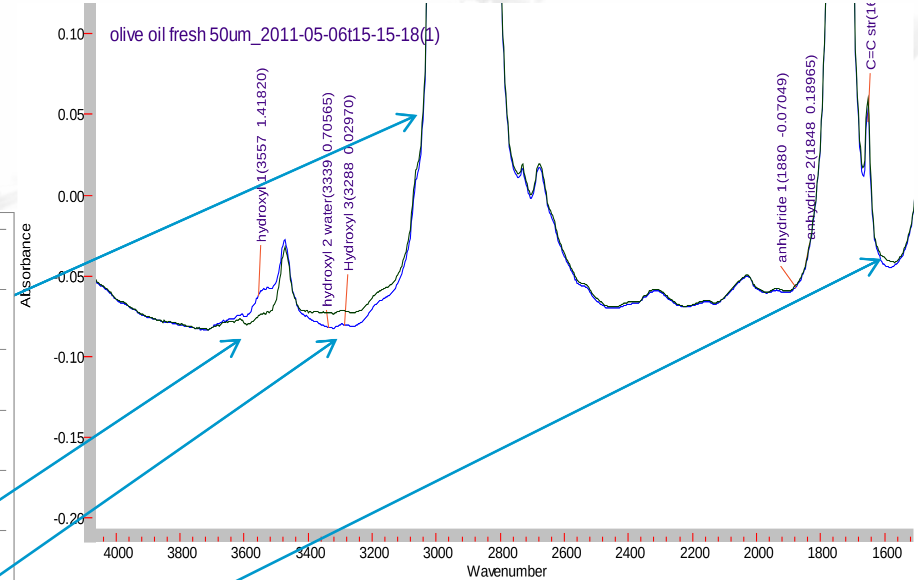


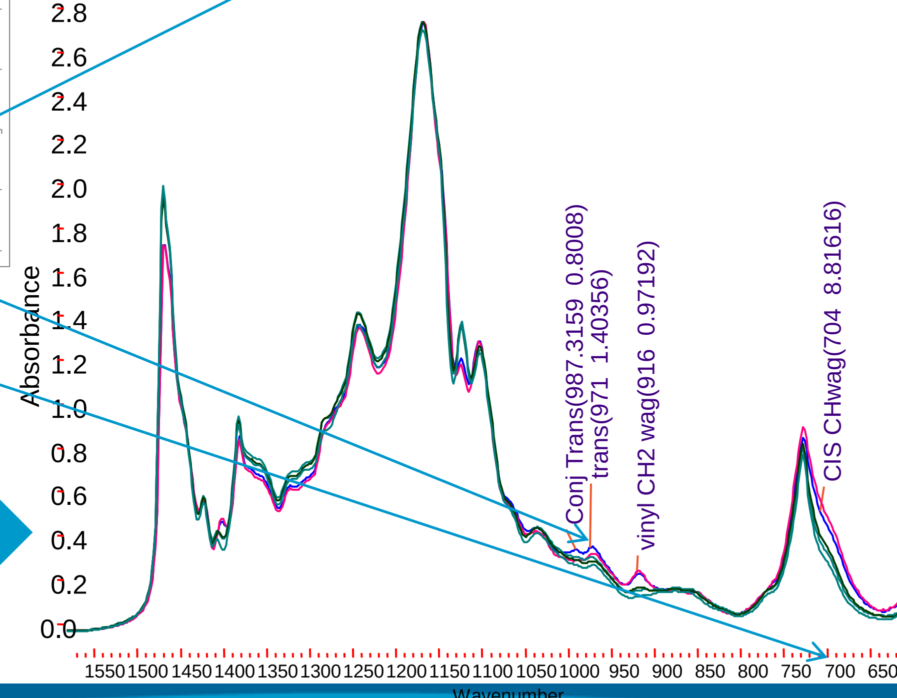
Peanut Oil

Olive Oil

# of Food Oils



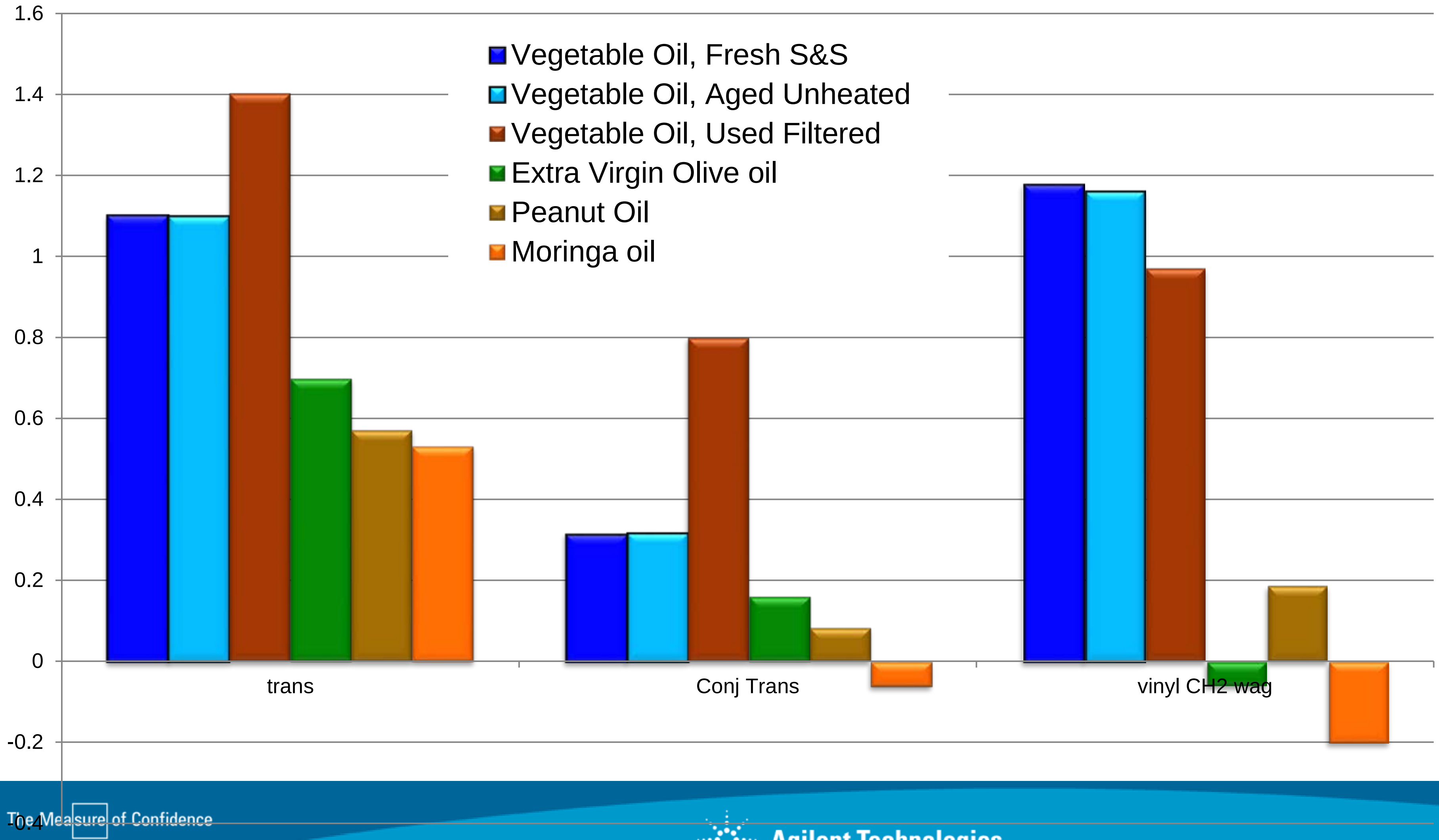




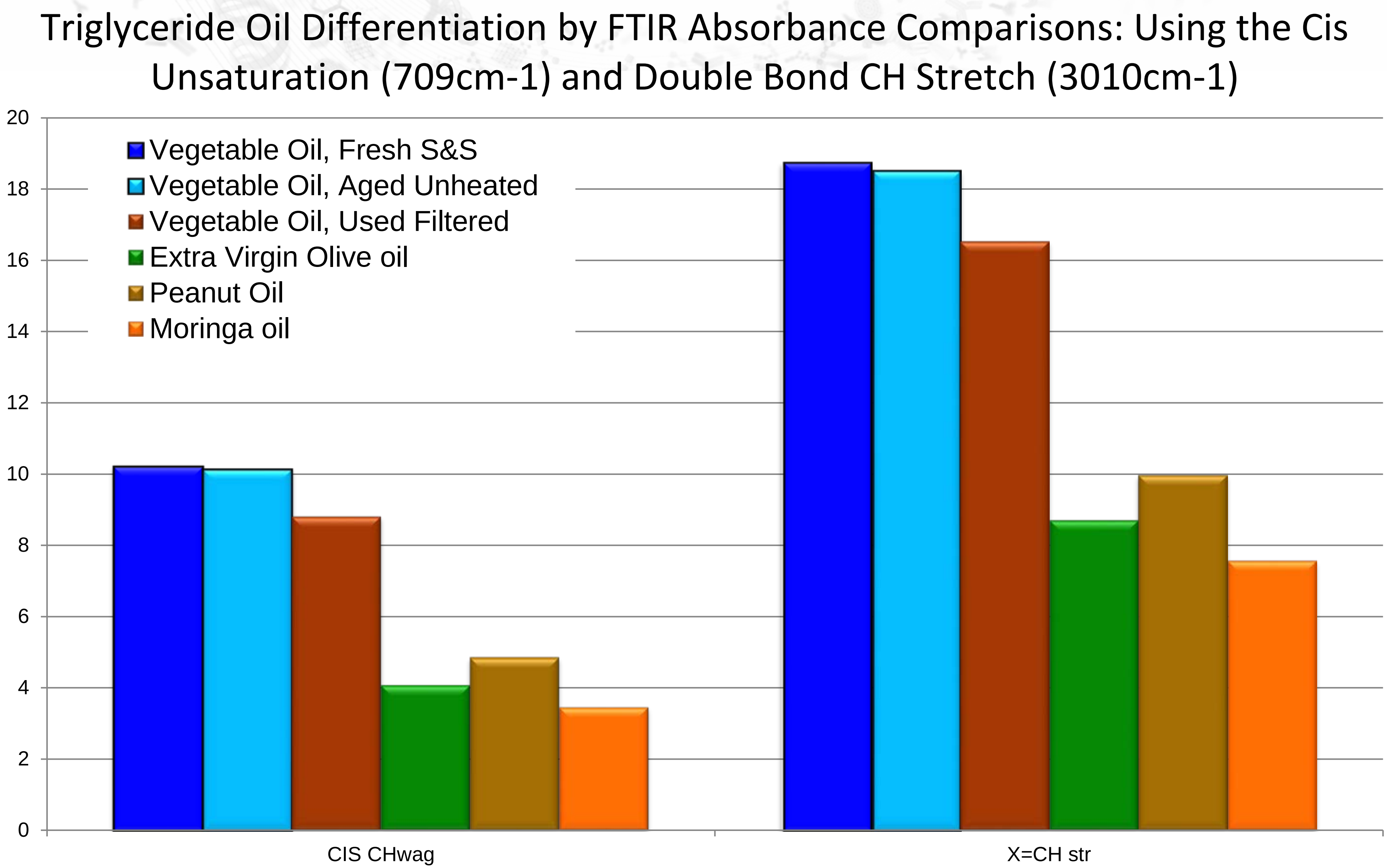
Three green spectra are olive oil, peanut oil, and moringa oil. The other two are vegetable oils, fresh (red) and used (blue).

Verification of Food Oils: Fatty Triglyceride Oils

Triglyceride Oil Differentiation by FTIR Absorbance Comparisons: Using the Trans Unsaturation (967cm-1), Conjugated Trans (988cm-1), and Vinyl CH wag (915cm-1)



Verification of Food Oils: Fatty Triglyceride Oils



Adulteration

Unethical food producers cut cost by diluting products with cheap alternatives

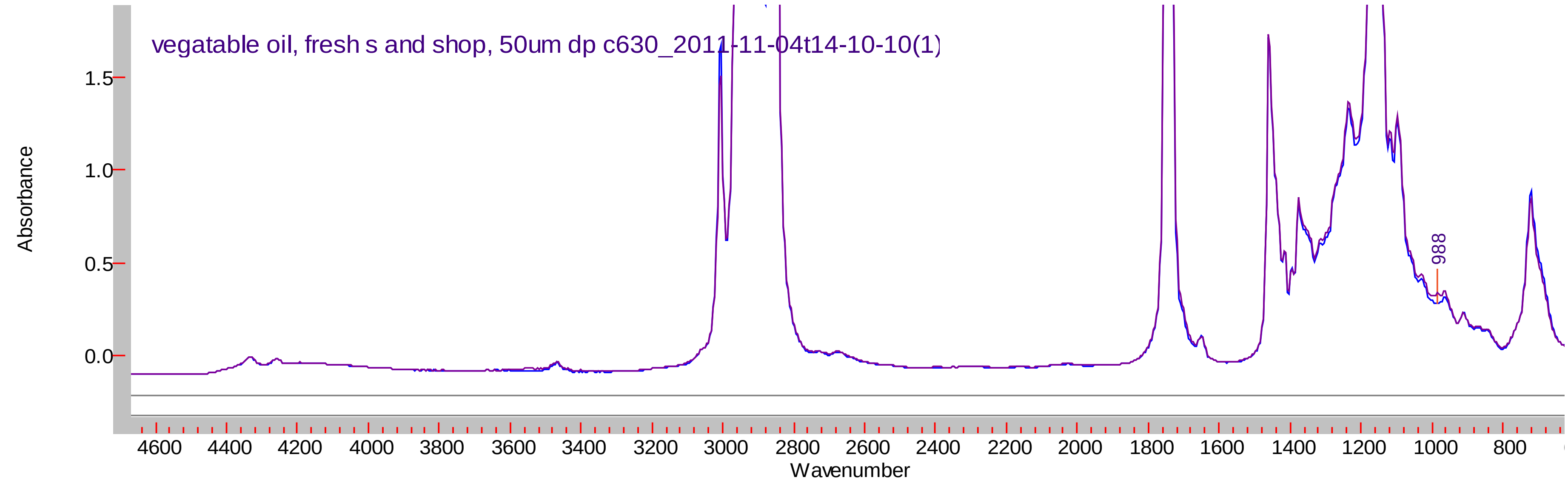
- Economically motivated
- Consumer satisfaction affected
- Adulterants typically % level

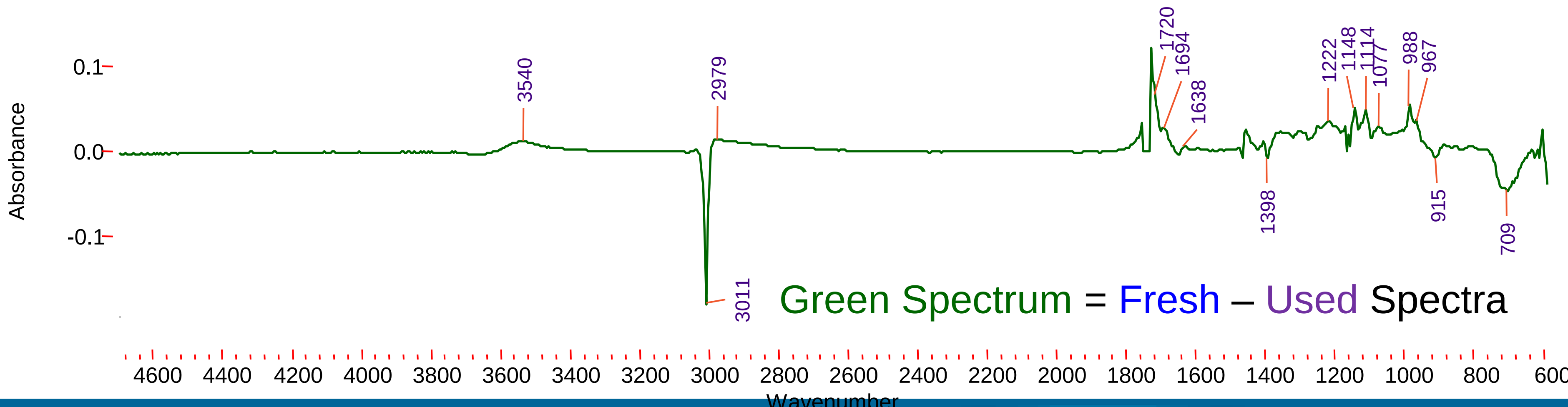
FTIR ideal for analysis

- Sensitive to small changes in chemistry
- Fast measurement
- Non-destructive

Food Oils - Vegetable Oil Qualification

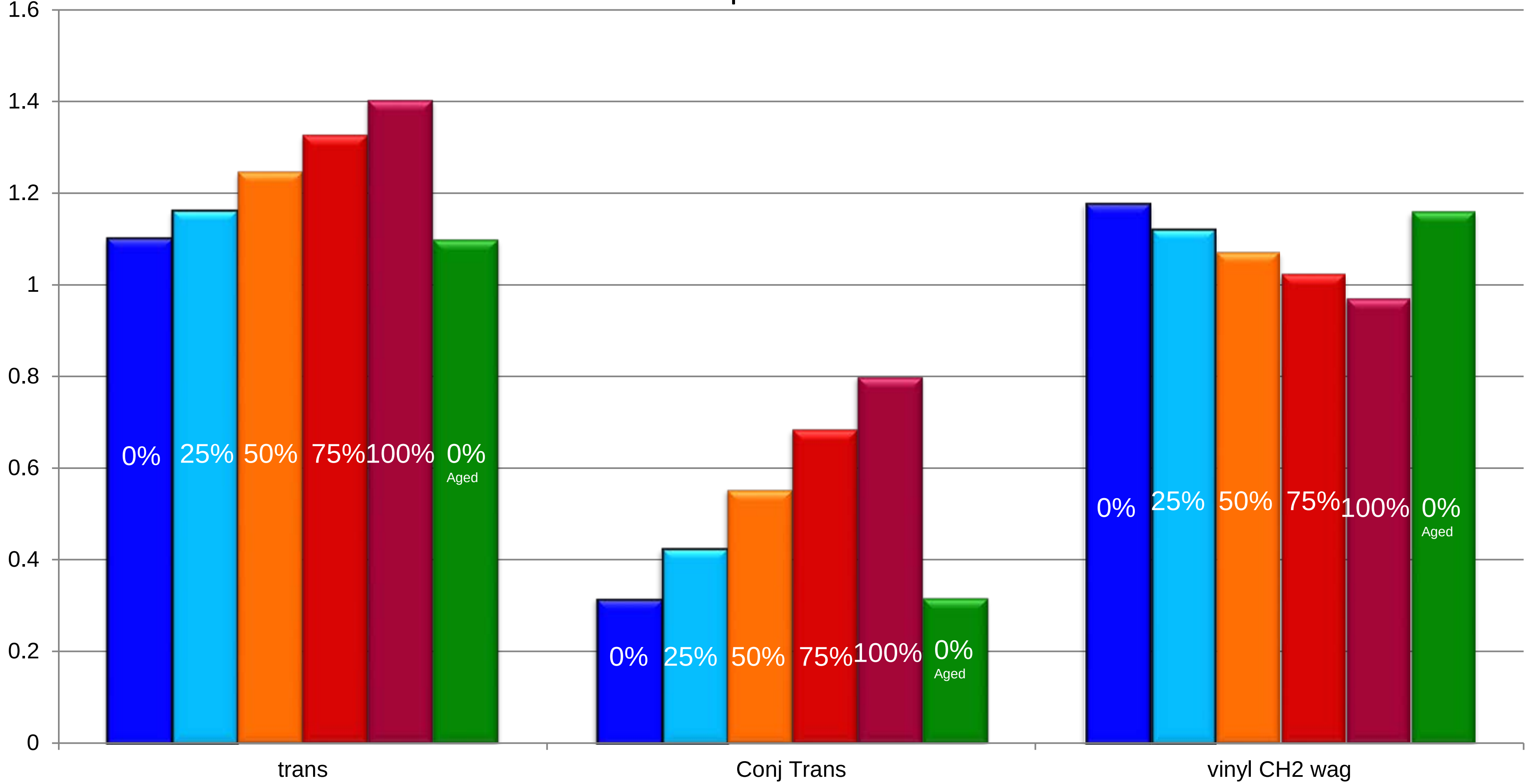
The FTIR subtraction spectrum (Green, bottom) of used (filtered) vegetable oil (Purple) minus the spectrum of fresh vegetable oil (Blue). Positive IR bands = created from oxidation or impurities --- Negative bands = consumed





New Vegetable Oil Adulteration with Used Veg Oil

Trans Unsaturation (967cm-1), Conjugated Trans (988cm-1), and Vinyl CH wag (915cm-1) absorbance bands. Fresh vegetable oil is diluted with increasing amounts of filtered used vegetable oil. The green column (0% used) is unheated and unused oil that has been stored (aged) for 6 months sealed at room temperature.



Globalization of the food chain presents difficult quality issues

Do you know the quality of your raw materials?

With coffee, tea, sugar, flour, and powdered dairy coming in from around the globe, how do you know what you are getting?

Coffee

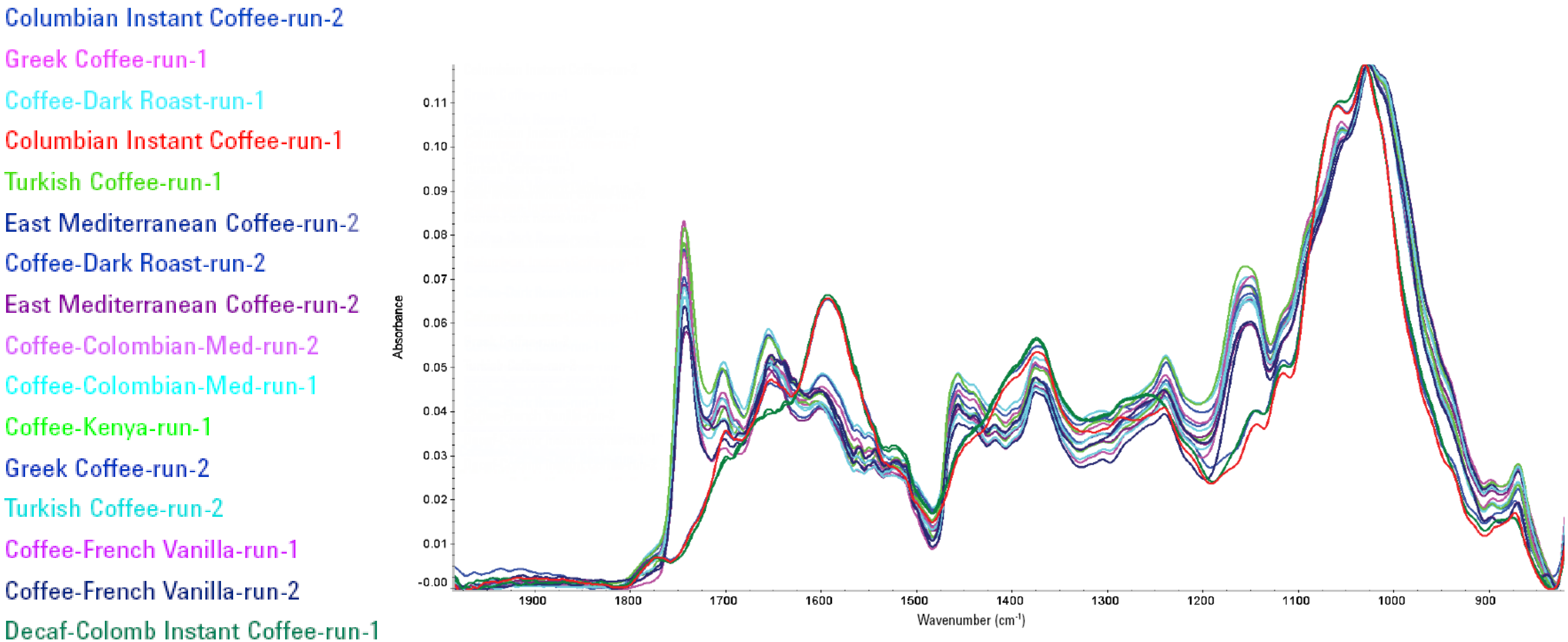
QA/QC of coffee using the Agilent Cary 630 ATR-FTIR analyzer

Authors: Zubair Farooq and Ashraf A. Ismail

Publication number: 5991-0783EN
Publication date: November 2012

Results for Coffee

Commercial ground-coffee brands were purchased and infrared spectra of the ground coffee samples were automatically stored in a spectral database created with the Agilent MicroLab FTIR software.



Infrared spectra of coffee measured by single reflection diamond ATR

Results for Coffee

Each ground-coffee brand exhibited characteristic infrared bands between 1800 and 800 cm-1.

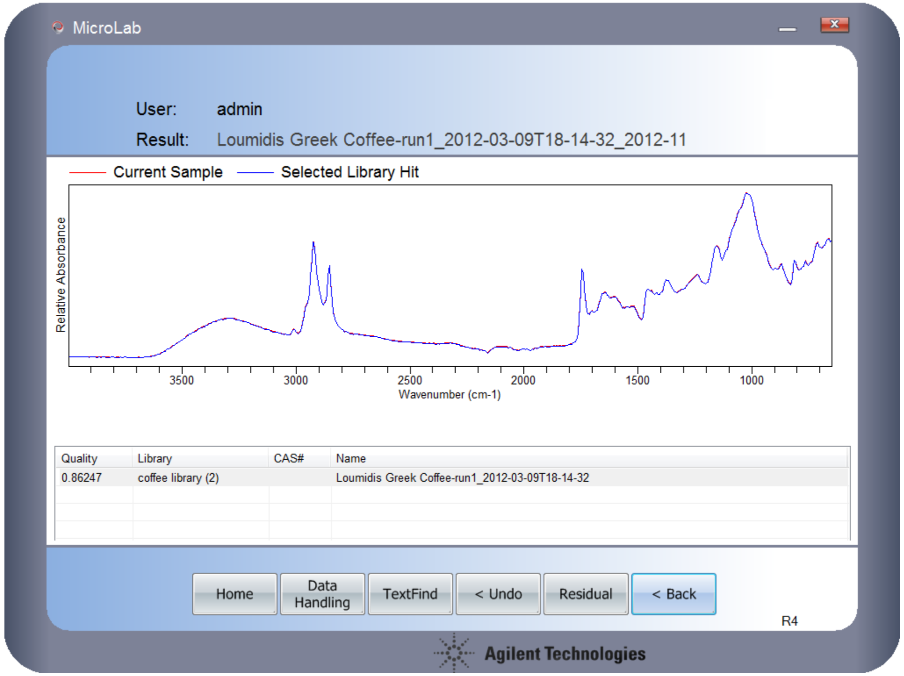
- The 1800 to 800 cm^{-1} region contains absorbance bands attributed to the stretching vibrations of C=O groups. These bands can be ascribed to organoleptic vinyl esters, lactones, esters, aldehydes, ketones, and acids present in the brewed coffee.
- C-H (methylene) bending (scissoring) absorptions occur between 1470 and 1430 cm^{-1} and can be assigned to lipids
- Bands appearing below 1400 cm^{-1} are generally referred to as the fingerprint region, as they are difficult to assign to specific functional groups. However, in this case, the strong absorptions between 1200 and 900 cm^{-1} can be assigned to C-O-H and C-O-C groups stemming from carbohydrate absorptions.

Results for Coffee

The entire spectral absorption region is used to differentiate coffee samples with varying chemical compositions.

ATR-FTIR spectra of different sample lots were acquired and their spectra compared to those already stored in the spectral library database.

In each case, the MicroLab software Search function correctly identified each coffee brand



Identification of an unknown coffee sample by the MicroLab FTIR software

Tea

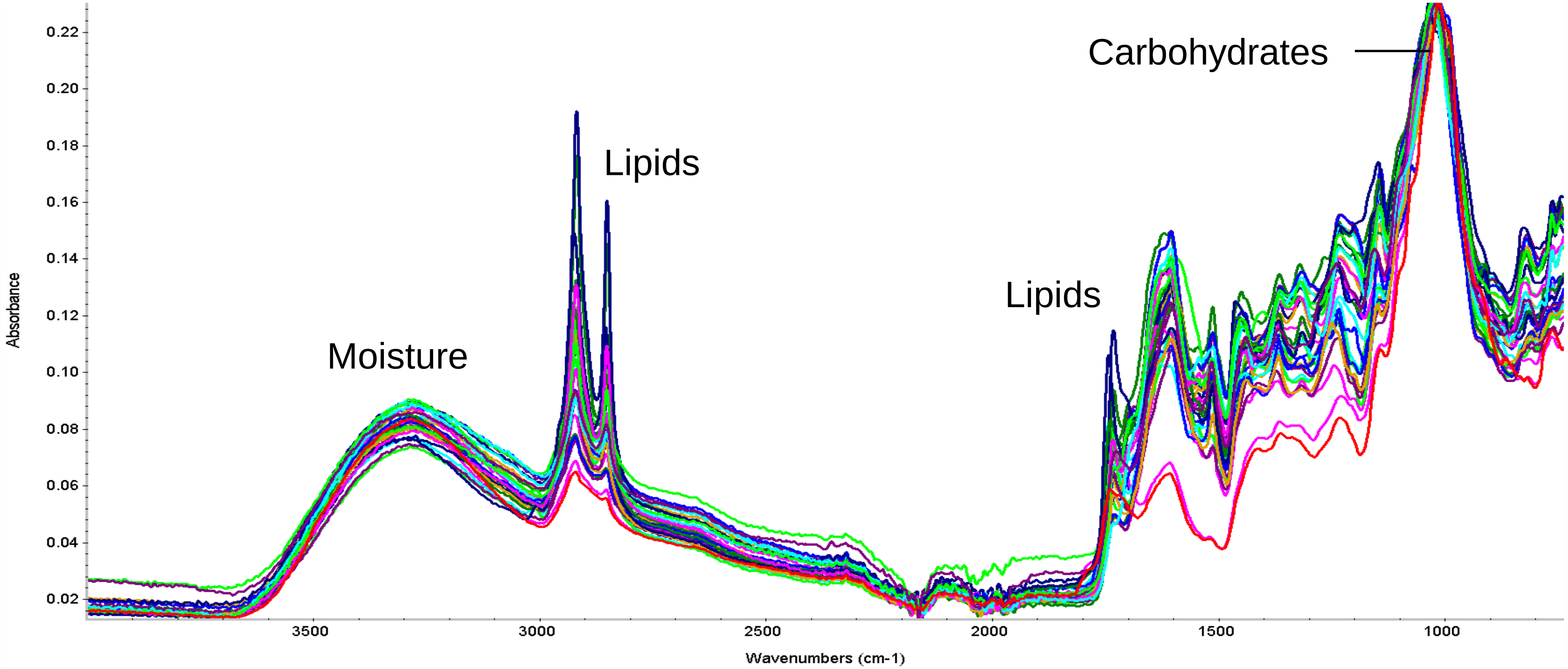
QA/QC of tea using the Agilent Cary 630 ATR-FTIR analyzer

Authors: Zubair Farooq and Ashraf A. Ismail

Publication number: 5991-0787EN
Publication date: November 2012

Results for Tea

Several different commercial tea brands were ground to produce uniformly-sized powders. The acquired spectra were stored in a spectral database using the MicroLab FTIR software.

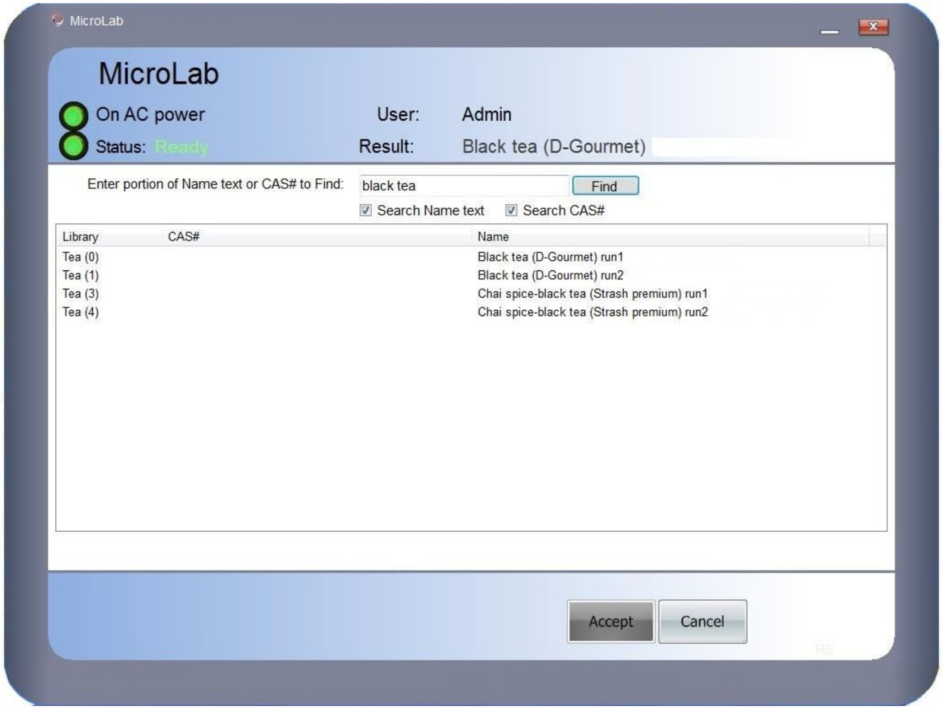


Agilent Cary 630 ATR-FTIR spectra of selected teas

Results for Tea

The spectral information between 3800 and 2600 cm^{-1} and 2000 and 600 cm^{-1} provides the means for differentiating between the different tea varieties.

These spectral regions contain absorption bands for all the major and minor organic components found in teas and therefore, all the spectral information within these regions plays an important role in distinguishing amongst teas with varying chemical compositions.



Example of the Agilent Cary MicroLab FTIR software for the correct identification of a new black tea sample

Sugar

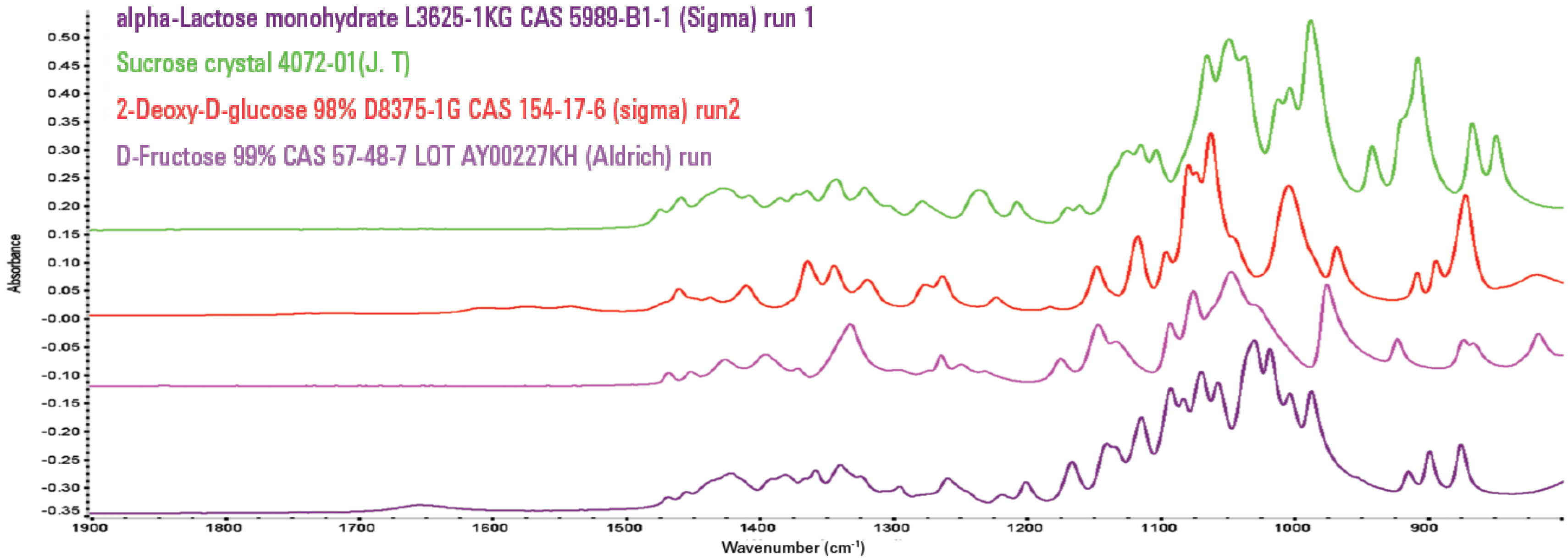
QA/QC of sugars using the Agilent Cary 630 ATR-FTIR analyzer

Authors: Zubair Farooq and Ashraf A. Ismail

Publication number: 5991-0786EN Publication

Results for Sugar

Most sugars are purchased as crystalline white powders. Examples are glucose, sucrose, lactose, fructose, maltose and xylitol. Protein-based sweeteners such as thaumatin, curculin and monellin are also used. Samples of various sugars were analyzed to build a reference database.

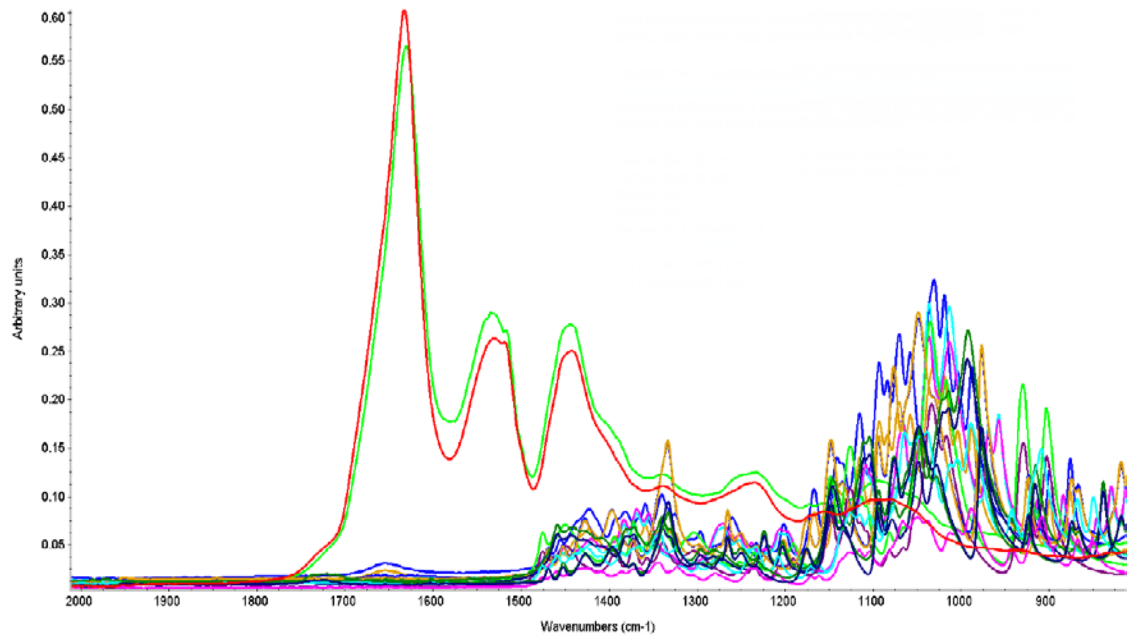


Offset infrared spectra of selected sugars

Results for Sugar

All carbohydrate-based sugars possess characteristic infrared absorptions between 1200 and 600 cm^{-1} attributed to C-O-H and C-O-C bonds. Protein-based sweeteners show unique absorption bands between 1700 and 1500 cm^{-1}, which can be attributed to the amide I and amide II absorption bands belonging to the proteins. Artificial sweeteners also possess unique absorptions depending on their chemical structure.

alpha-Lactose monohydrate L3625-1KG CAS 5989-B1-1 (Sigma) run2
alpha-Lactose monohydrate L3625-1KG CAS 5989-B1-1 (Sigma) run1
Thaumatin from Thaumatococcus daniellii T638 (Sigma) run1
Thaumatin from Thaumatococcus daniellii T638 (Sigma) run2
D-Mannose 99% mixture of anomers CAS 3458-28-4 LOY PY 03208PY (Aldritch) run2
D-Mannose 99% mixture of anomers CAS 3458-28-4 LOY PY 03208PY (Aldritch) run1
D-Xylose 99%+ W36060-0 KW03110LT (Aldritch) run2
D-Xylose 99%+ W36060-0 KW03110LT (Aldritch) run1
Fructose (Aldritch) run1
Fructose (Aldritch) run2
Glucose run1
Glucose run2
Sucrose crystal 74771 (Merck) run1
Sucrose crystal 74771 (Merck) run2
d (-) Levulose (NBC) run1
d (-) Levulose (NBC) run1

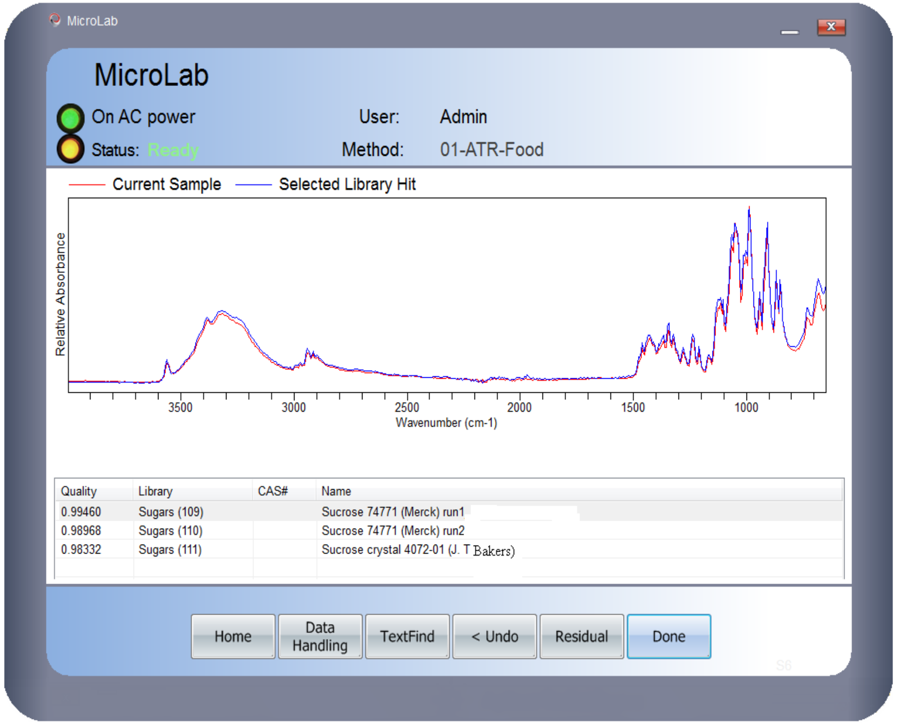


The strong infrared absorption band located between 1700 and 1600 cm-1 is attributed to the amide I band of protein sweeteners

Results for Sugar

Due to the similarity in their color, odor, texture and general appearance, it is very difficult for processors to authenticate the sugar identity prior to its addition to a formulation.

When a new or unknown sweetener is received, its spectrum can be compared to those stored in the database of previously recorded sweeteners, and its identity can be correctly established by the MicroLab FTIR software



Identification of an unknown sugar sample by the MicroLab FTIR software

Flour

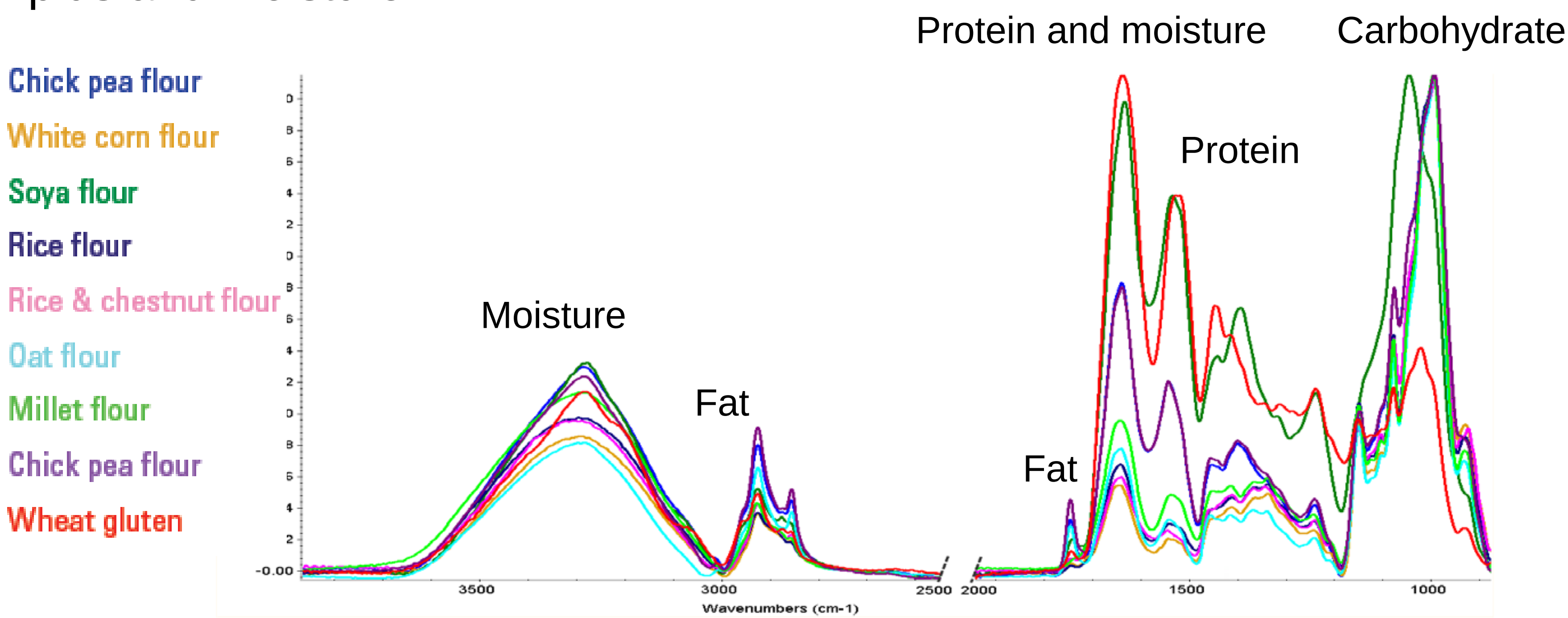
QA/QC of flours using the Agilent Cary 630 ATR-FTIR analyzer

Authors: Zubair Farooq and Ashraf A. Ismail

Publication number: 5991-0785EN
Publication date: November 2012

Results for Flour

- Samples of flours and flour products were obtained from different suppliers.
- Infrared spectra for selected flour samples show the infrared bands characteristic of major flour components such as proteins, carbohydrates, lipids and moisture.

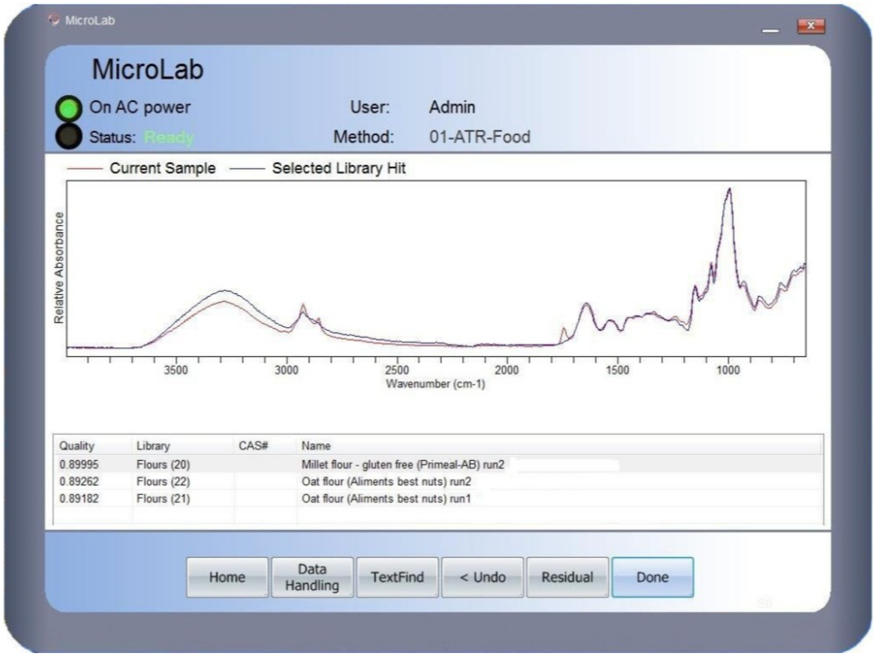


Overlapped infrared spectra of selected flour powders

Results for Flour

A spectral database can be quickly created on-the-fly in MicroLab PC. A sample can then be identified immediately from its infrared spectrum .

The spectral similarity among the infrared spectra of incoming new materials and previously recorded samples is particularly valuable in tracking batch-to-batch variability from the same or different vendors.



Agilent MicroLab FTIR software correct identification of an unknown flour sample

Dairy Powder

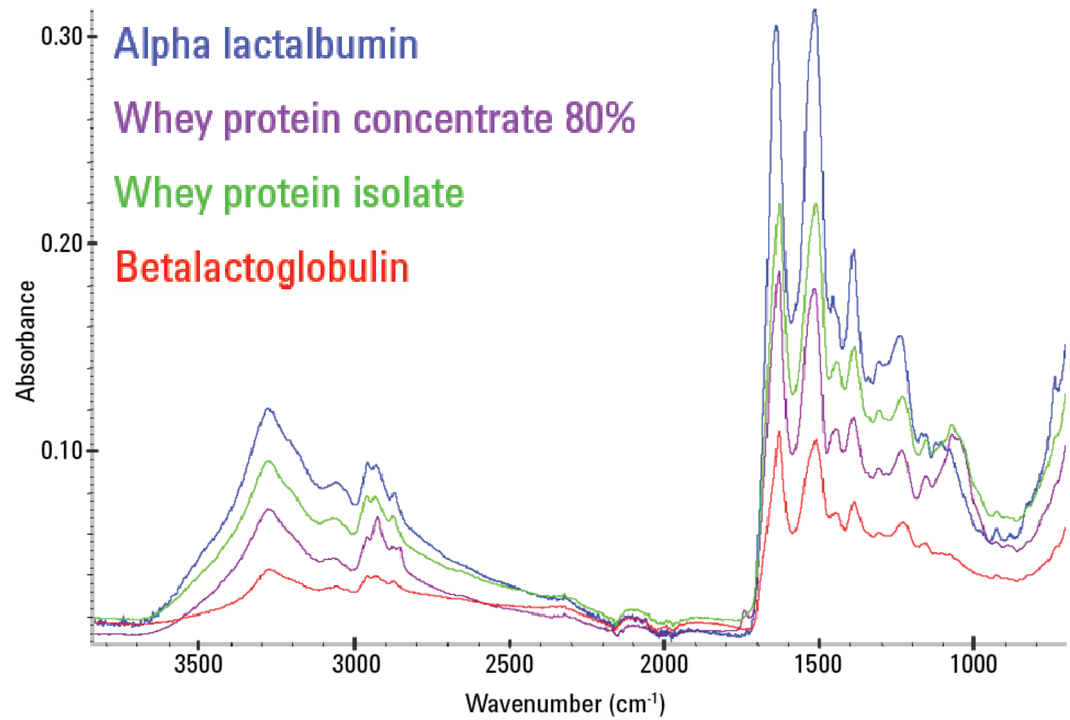
QA/QC of dairy powders using the Agilent Cary 630 ATR-FTIR analyzer

Authors: Zubair Farooq and Ashraf A. Ismail

Publication number: 5991-0784EN
Publication date: November 2012

Results for Dairy Powder

- Milk protein powder samples including α-lactalbumin, β-lactoglobulin, glycomacropeptide, milk protein concentrate, WPI, WPC, caseins and caseinates were obtained from different suppliers.
- Spectra of different lots of dairy powders were then recorded and treated as ‘unknowns’.

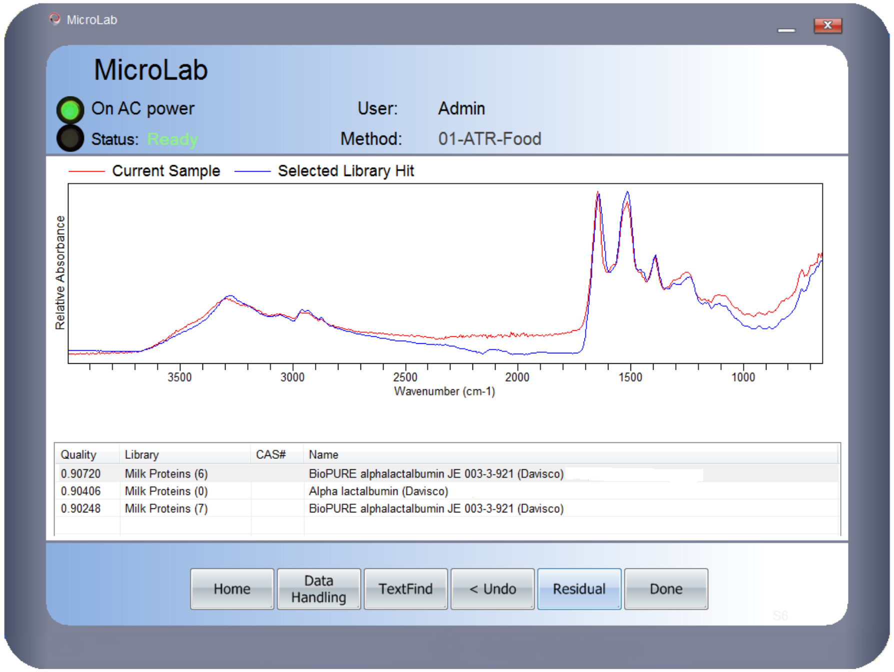


Infrared spectra of selected dairy powders

Results for Dairy Powder

The difference between whey protein concentrate (WPC) and the other three proteins can be clearly seen in the presence of additional bands between 1300 and 900 cm^{-1} attributed to the presence of lactose in WPC.

The differences among the three other proteins are harder to distinguish by the naked eye. All four protein types can be immediately characterized and differentiated with the built-in spectral analysis MicroLab FTIR software.



Correct identification of an unknown dairy powder as α-lactalbumin by the Agilent MicroLab FTIR software

FTIR Applications

- The robust, light-weight and compact **Agilent Cary 630 FTIR** with ATR sampling interface is the ideal technology to verify the identity and chemical composition of food ingredients, eliminating the need for onerous sample preparation or extraction procedures.
- Analytical results are available in seconds with minimal operator training.
- The instrument is ideal for use at the receiving dock, in the production line, in the QA/QC laboratory, or for onsite verification at the ingredient supplier depot.
- *It is the extreme ease of use and robustness that truly puts the Cary 630 ahead of anything else on the market.*

To solve the toughest problems, we will partner with some of the world's best minds

People & Expertise

Food safety leaders

Global food industry organizations

Major laboratories

Leading government agencies

Prestigious universities

Application pace-setters

Global leaders and emerging researchers in specific application areas

Technology Innovators

Government/academic labs that are leading the way in novel food safety technologies

Only Agilent has the ***chemical*** and ***biological*** technology/measurement capabilities to support a comprehensive food safety program

Only Agilent is prepared to put ***time***, ***money***, and ***talent*** toward finding and developing new food safety applications

Only Agilent has created a collaborative process for meeting ***current*** and ***future*** food safety challenges

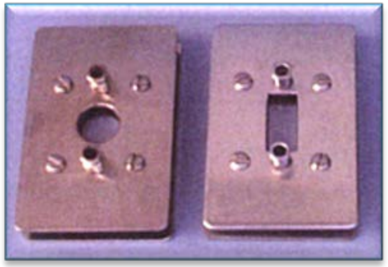
TRANSMISSION SPECTROSCOPY OF LIQUIDS USING AGILENT'S DIALPATH TECHNOLOGY

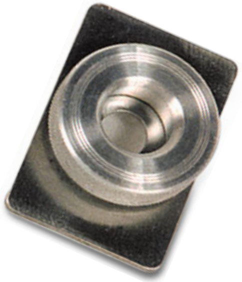
ELIMINATING THE CLASSIC TRANSMISSION CELL

Current Analysis of Liquids by FTIR

For longer pathlength measurements (>30 microns - lower concentration solutes; IR transparent solutions)

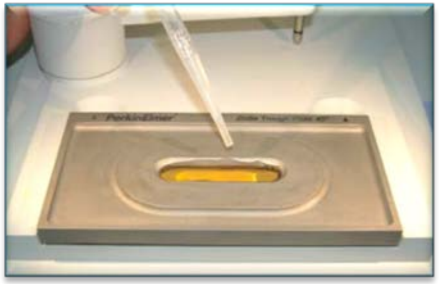
- Fixed pathlength transmission cells
- Demountable, variable pathlength transmission cells





For shorter pathlength measurements (<30 microns – higher concentration solutes; IR opaque solutions)

- Internal reflection



No real change in the past 30 years on how liquids are measured via mid IR

Beers Law - Our Favorite

Beer's Law (Absorbance Law): IR absorbance is directly proportional to concentration

A=abc

A is Absorbance, b=thickness, c=concentration, a=absorptivity constant

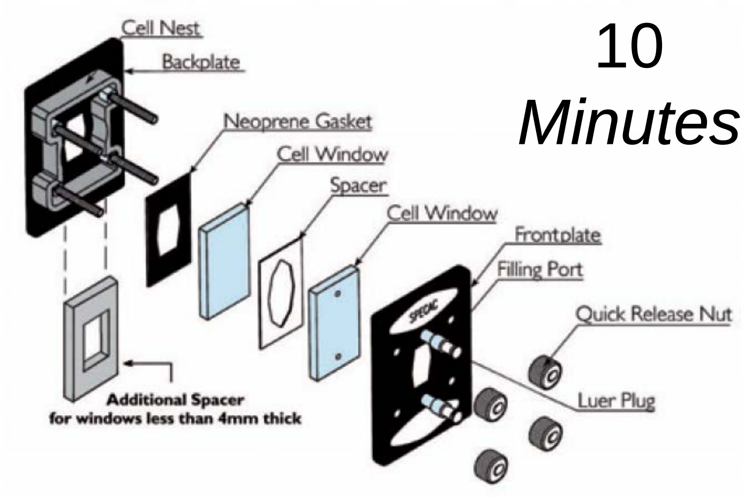
FT-IR measures each peak separately

FT-IR can measures all compounds in a mixture at the same time (one spectrum can yield concentrations for dozens of components)

Liquid Samples

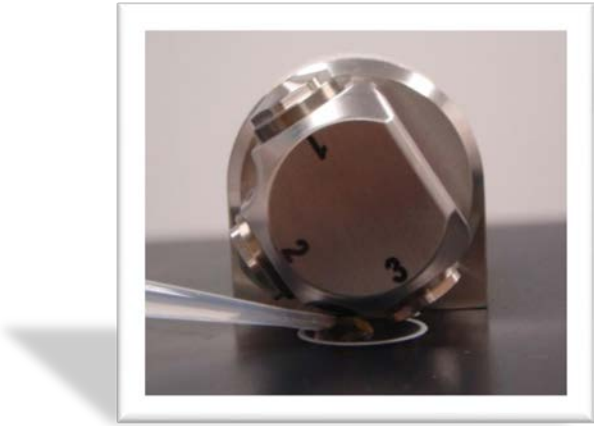
Anyone who has measured liquids with traditional cells will be ***AMAZED*** at how much faster and easier it is to measure liquids with the DialPath or TumblIR. This patented technology is a ***HUGE*** time saver in the lab and is available only from Agilent.

Traditional liquid cell



10
Minutes

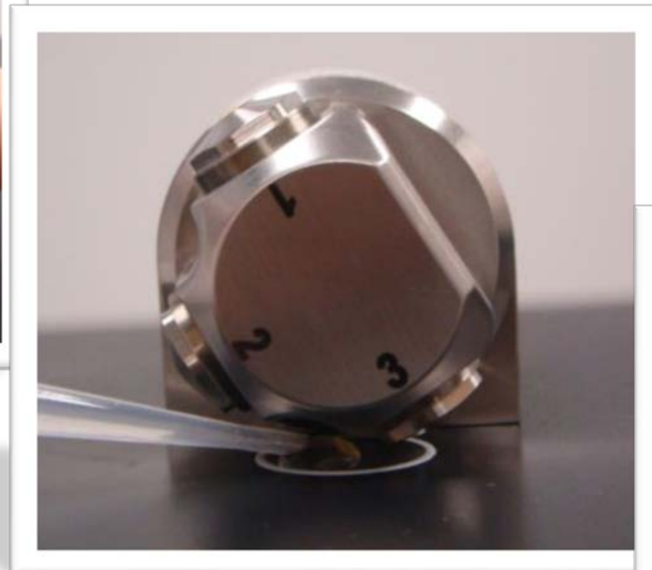
The New 'Agilent' Way



10
Seconds

Revolutionary technology for Liquids Analysis

"Dialpath"



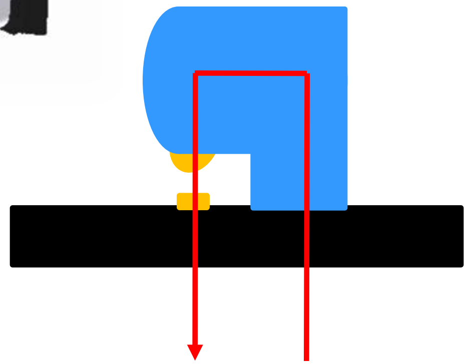
Proprietary Transmission Techn

As quick and easy to carry out longer pathlength transmission measurements as ATR makes shorter pathlength analysis

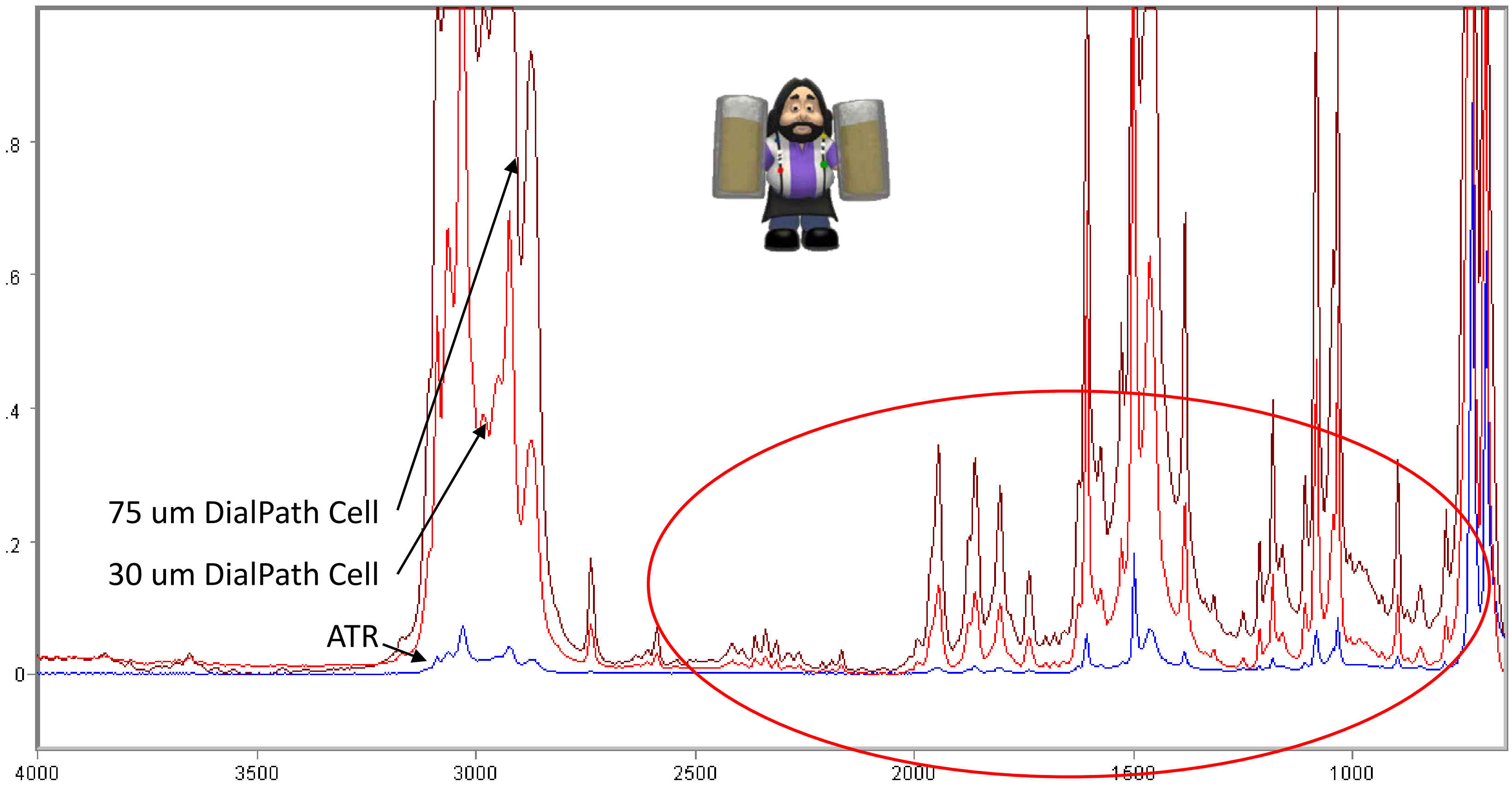
- DialPath: Choice of three factory calibrated, fixed pathlengths can be selected in seconds
- TumblIR: One dedicated Pathlength
- Covers pathlengths that ATR cannot
- No spacers, windows, or syringes needed
- No fringing

Sample introduction and sample cleaning is simple – takes seconds

- Compatible with samples having a wide range of viscosities
- Eliminates the need to heat samples to reduce viscosity

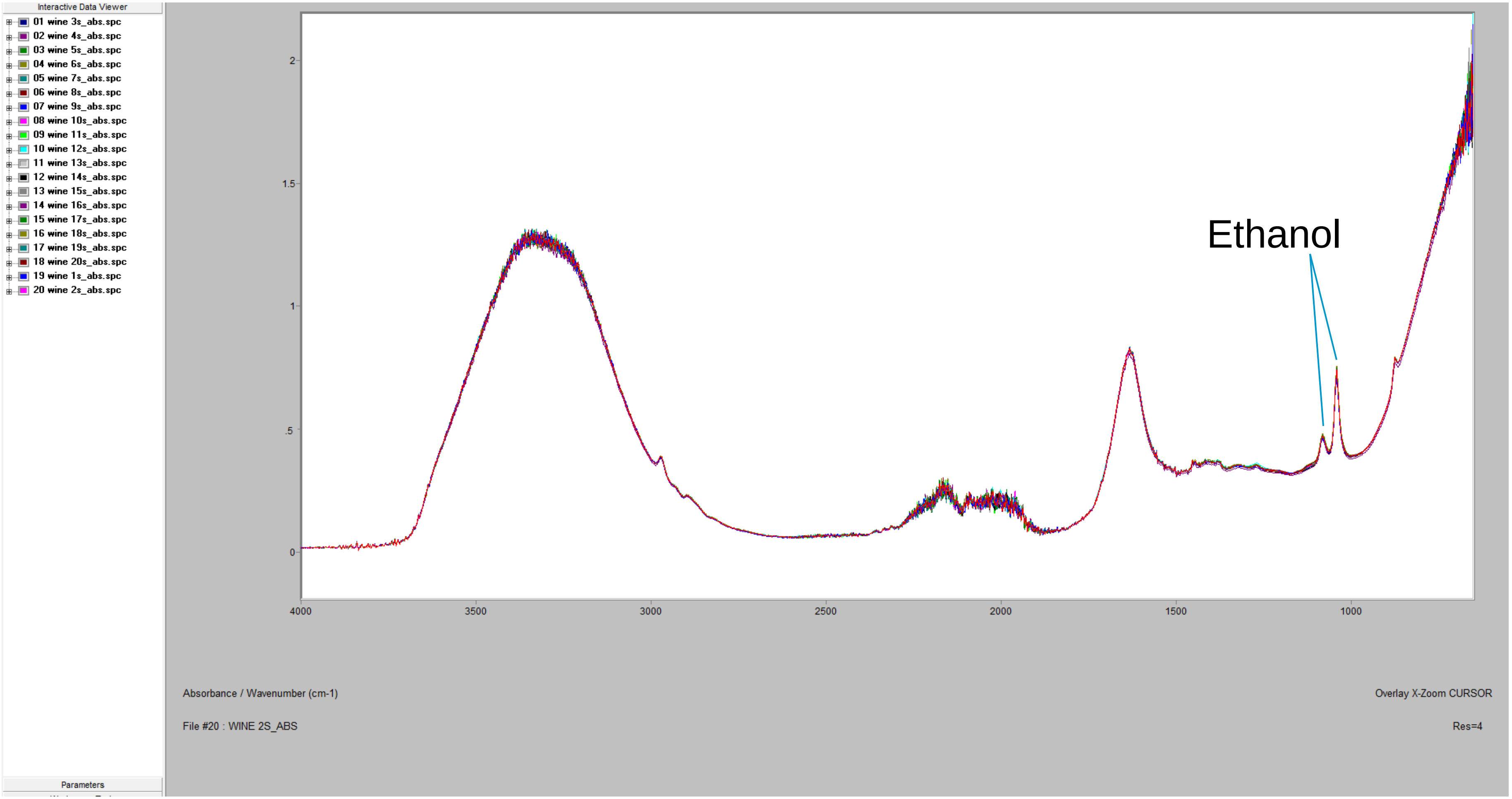


Increased Sensitivity v ATR

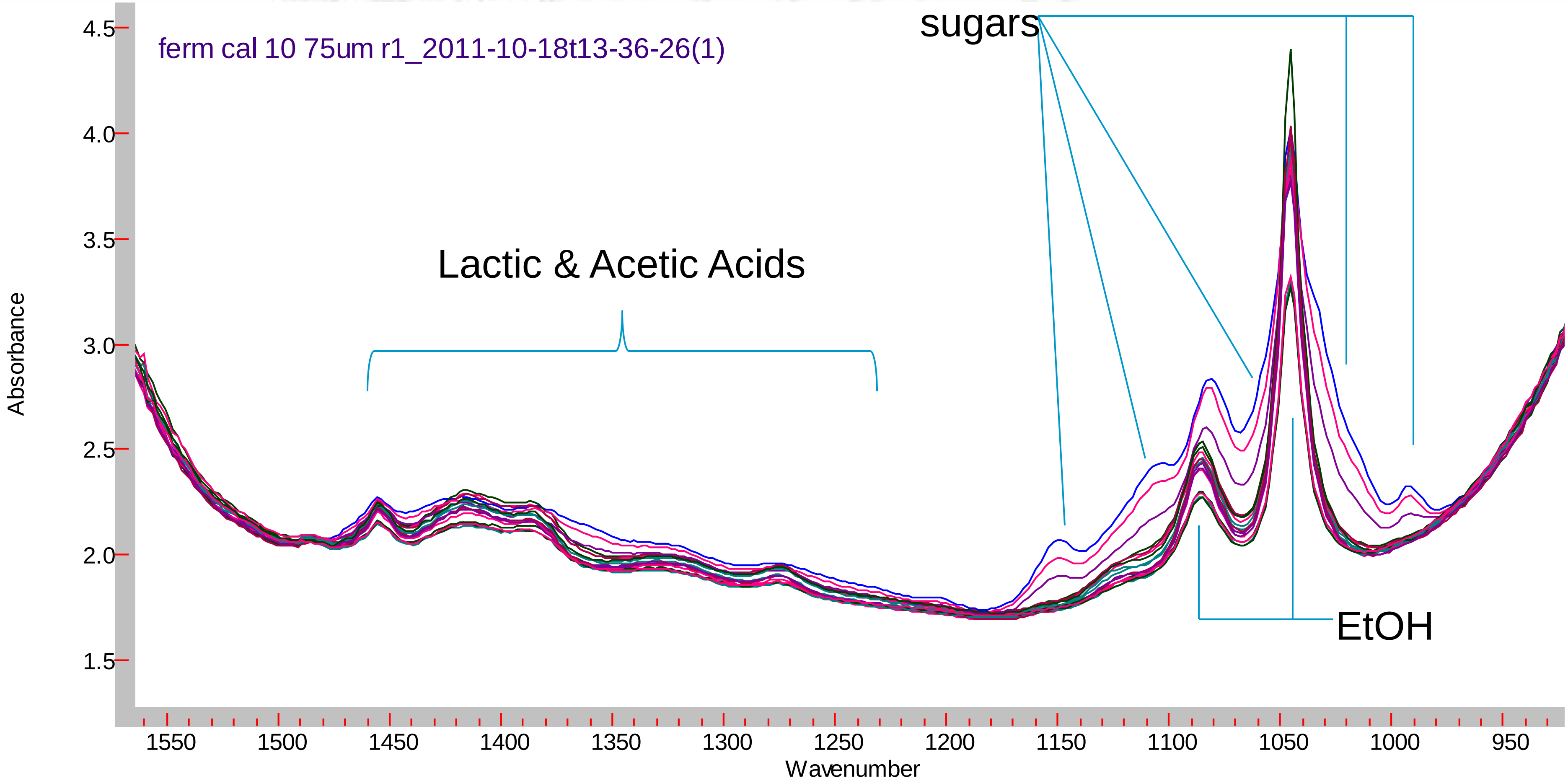


Looking at content of drinks

Full spectrum overlay of 20 different wines, multibounce ATR Agilent 5500a

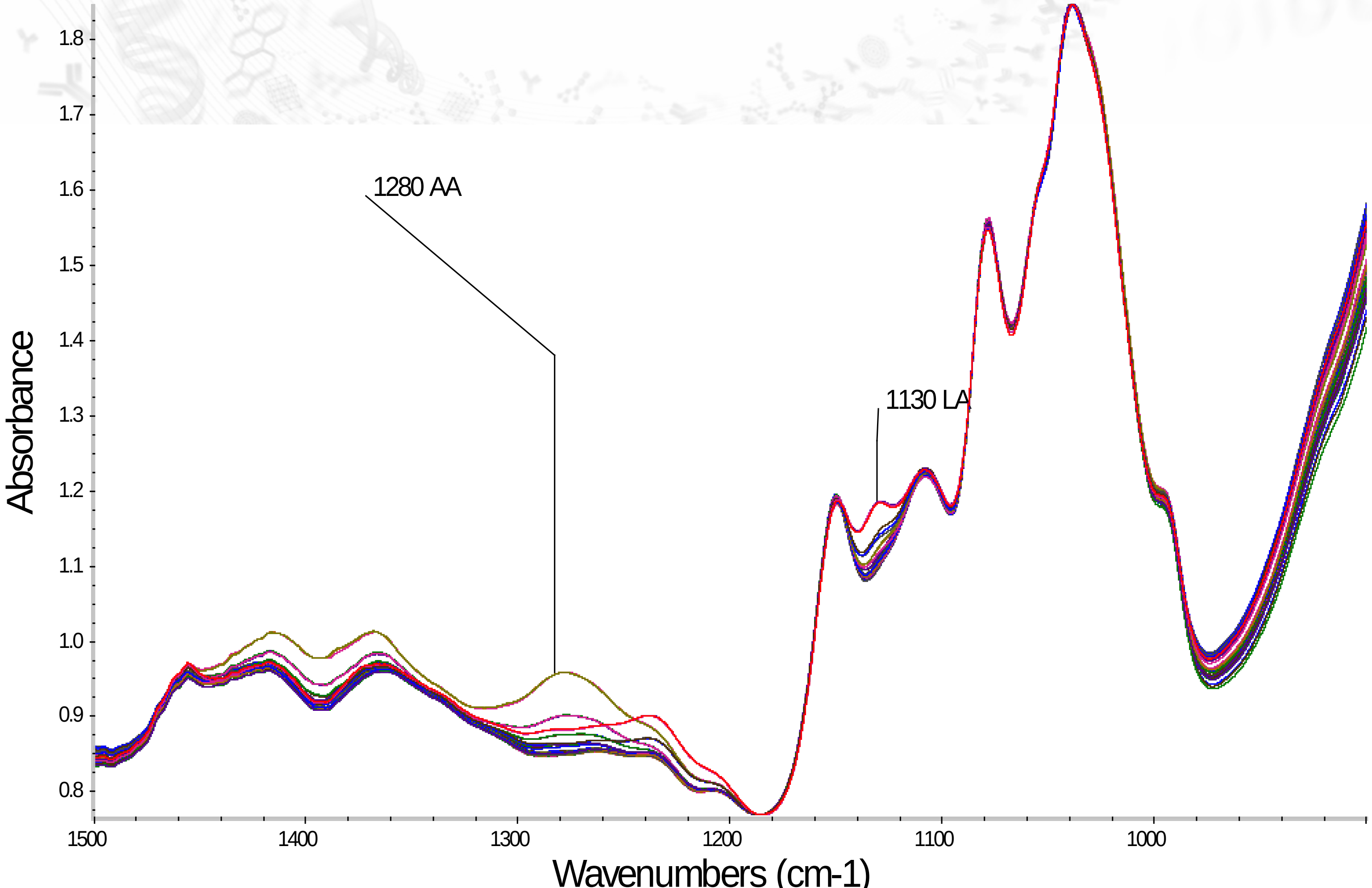


FTIR spectral overlay, fingerprint 1550-900cm-1 region, of fermentation at different stages in the fermentation process. The spectra are collected on a Dialpath 5500t with a 75um pathlength. The ethanol and sugar bands are shown.

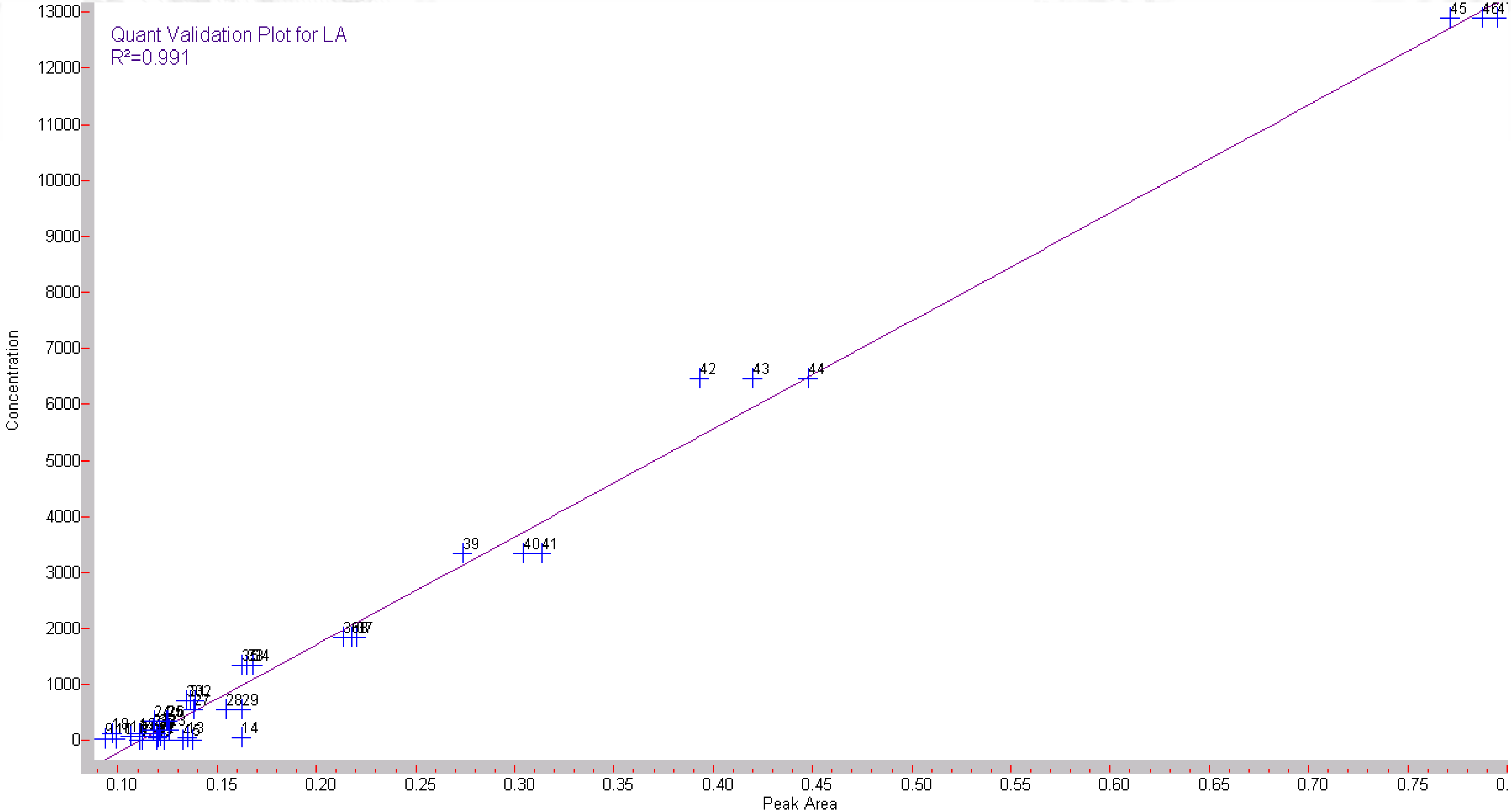


Background

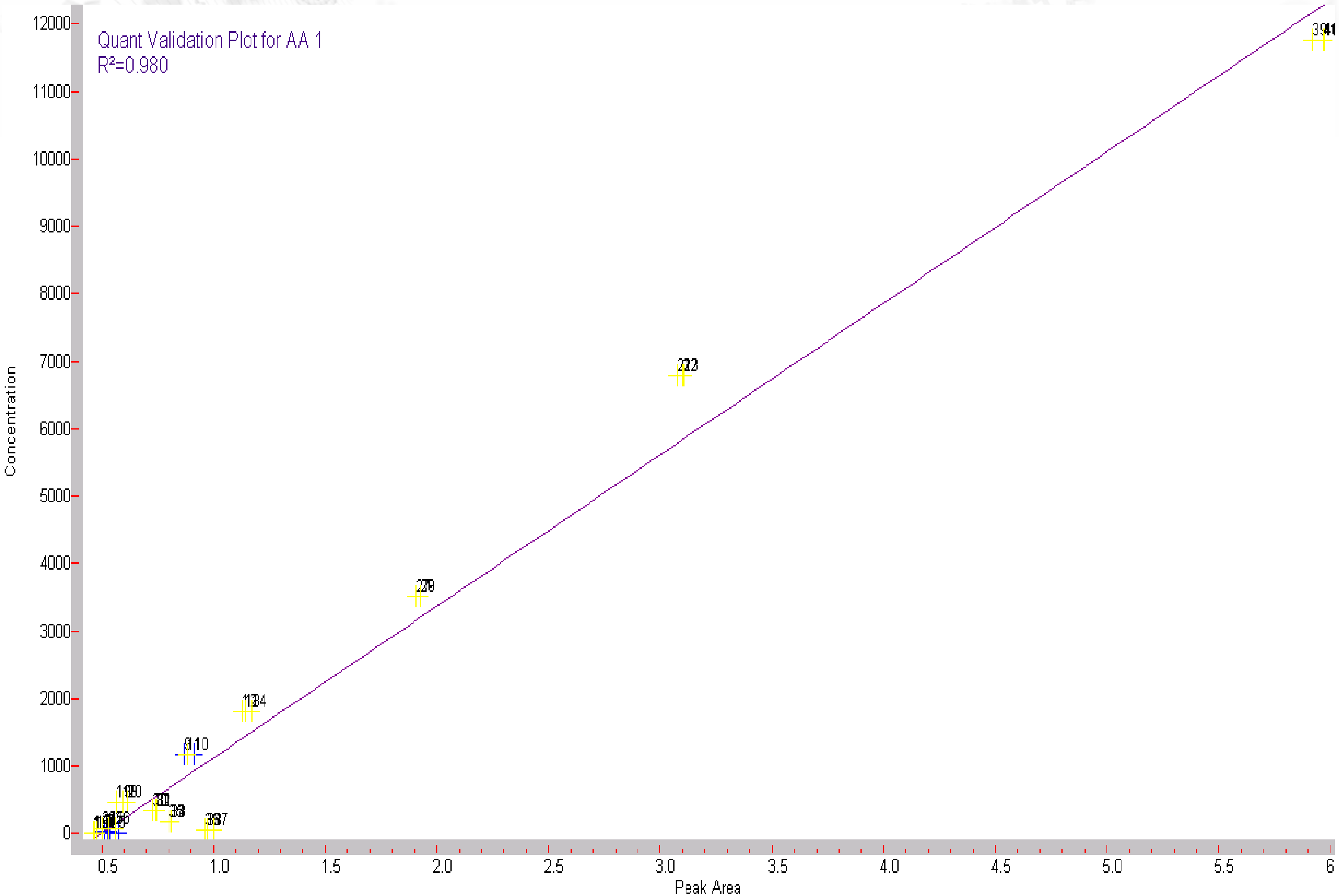
Work on customer samples that wanted quick way to monitor the acid ingredients in their fermentation matrixes which along with high sugars decreased ethanol formation by. Goal was to see if a quick FTIR analysis could give them quick feedback to help control the fermentation process saving them a significant amount of time and money by augmenting the current analytical method (e.g. titration and chromatography).

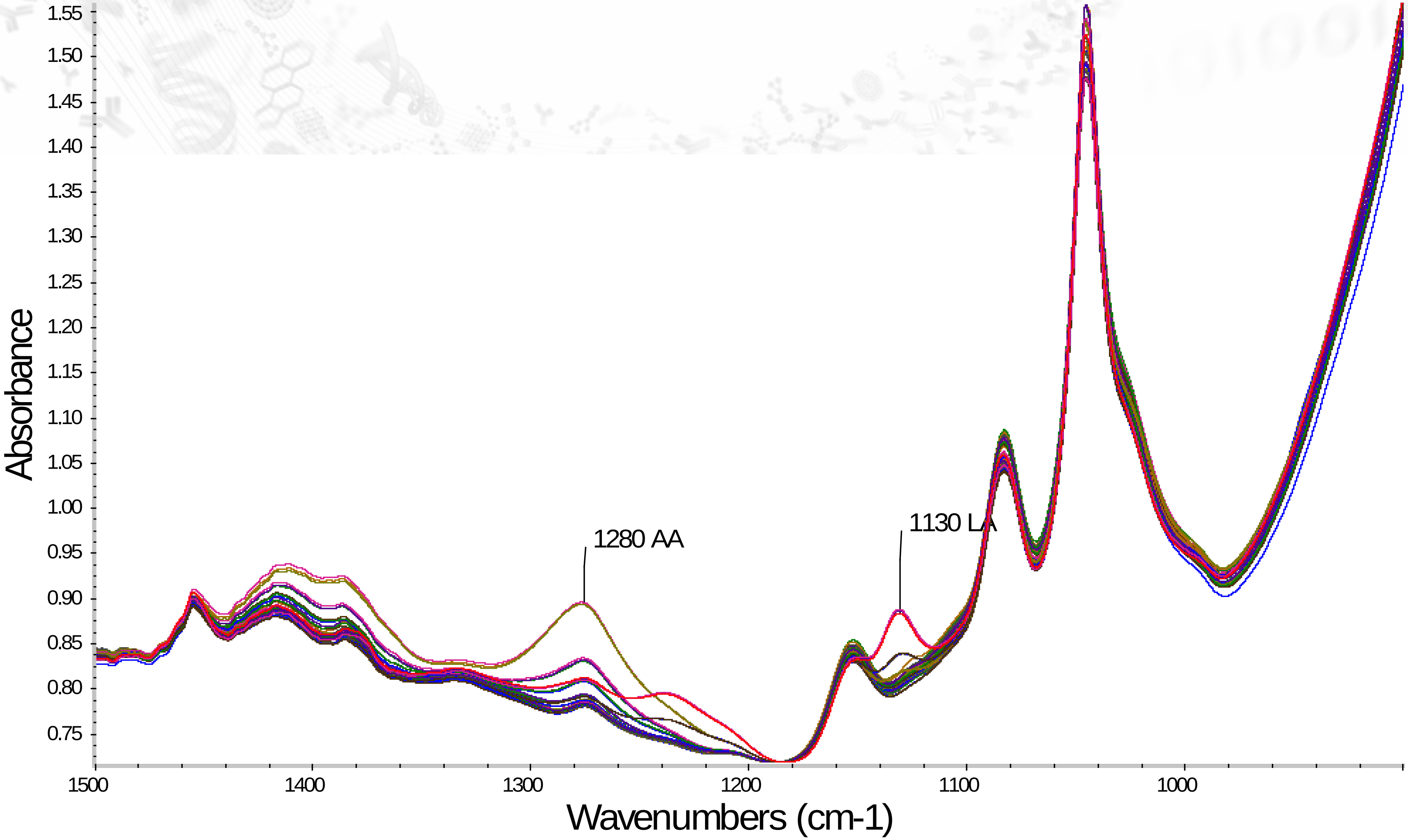


Transmission (i.e. Dialpath) quant Validation plot s of the two acid component in matrix **SB1 .** Overlaid spectra with marked index bands (1500-900 cm-1) with the marked index features; LA validation plot and AA validation plot

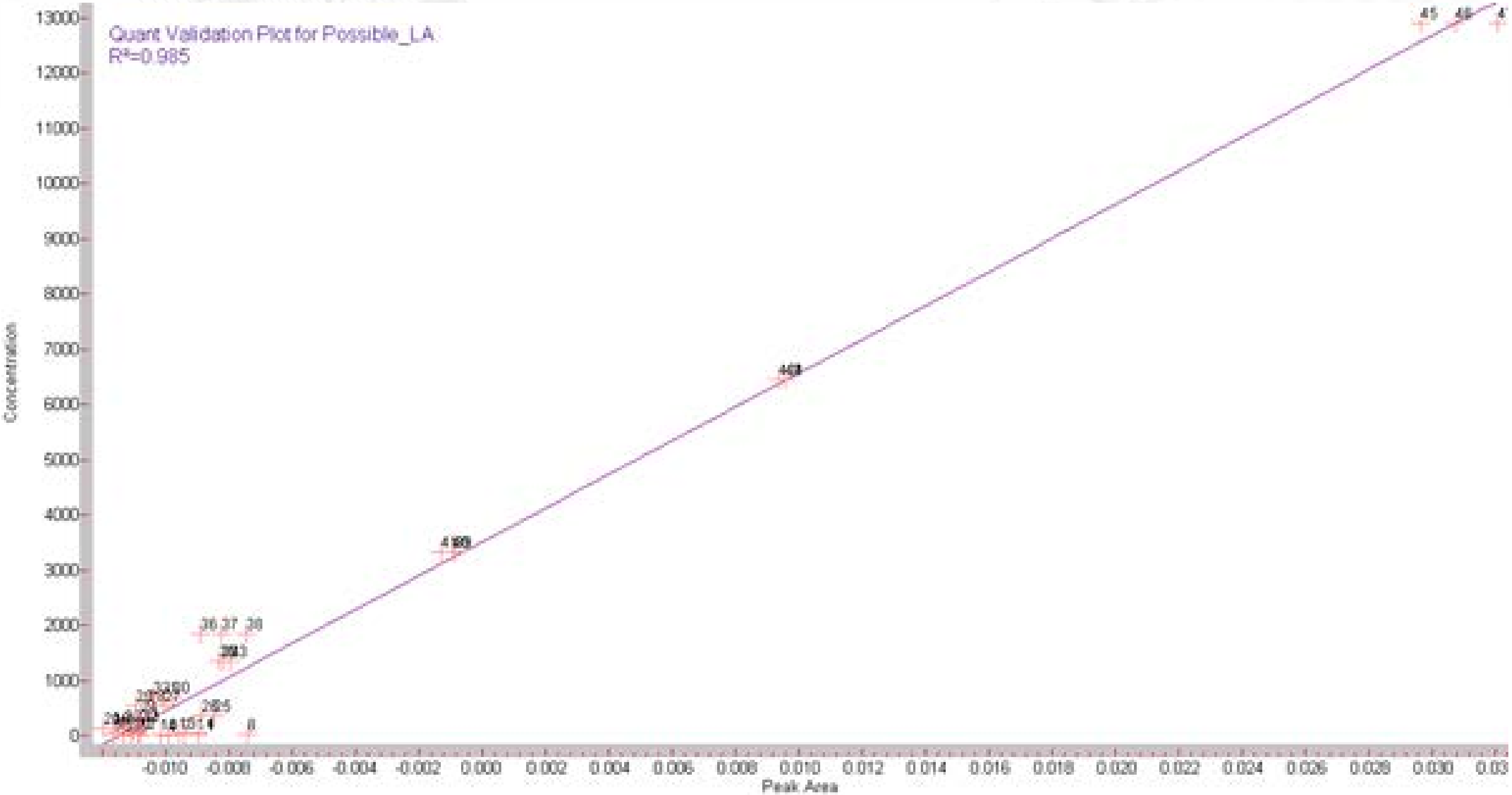


It should be noted that the area integration of the indexing bands were used to create the validation plots. For LA index, the local peak position was near 1130 (not like 1150 in reference spectra), which could be associated with the different concentration level (e.g. different hydrogen-bonding) and the matrix interference.

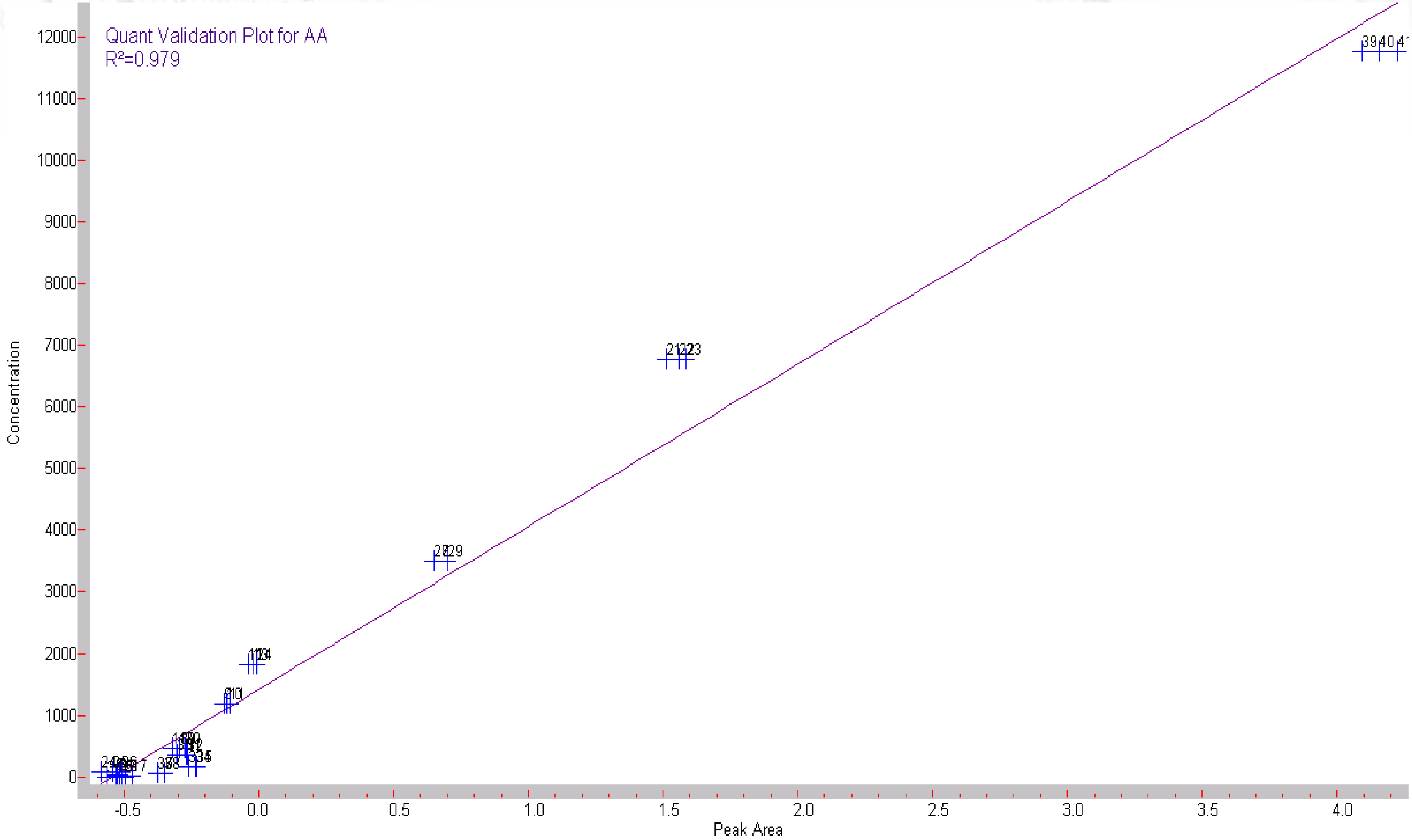

Quant Validation Plot for AA 1
R²=0.980
Concentration
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6
Peak Area

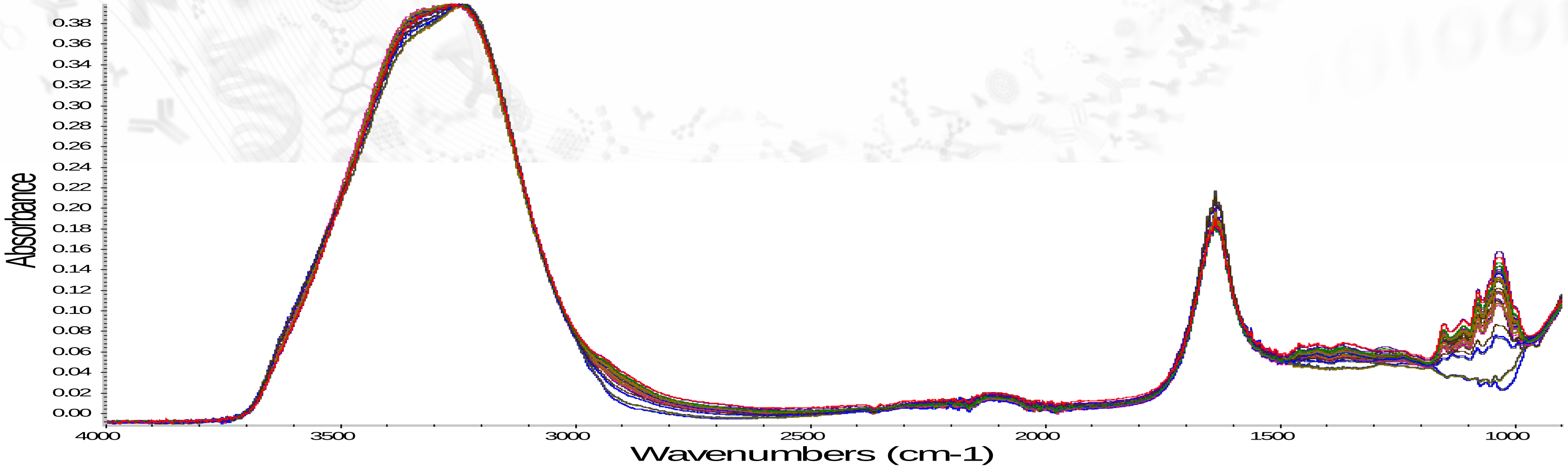


Transmission (i.e. Dialpath) quant Validation plot s of the two acid component in matrix **SB2.** **(A)** Overlaid spectra with marked index bands (1500-900 cm-1); **(B)** LA validation plot (all data used) and **(C)** AA validation plot (two samples #14 and 15 were excluded due to interference of high LA).

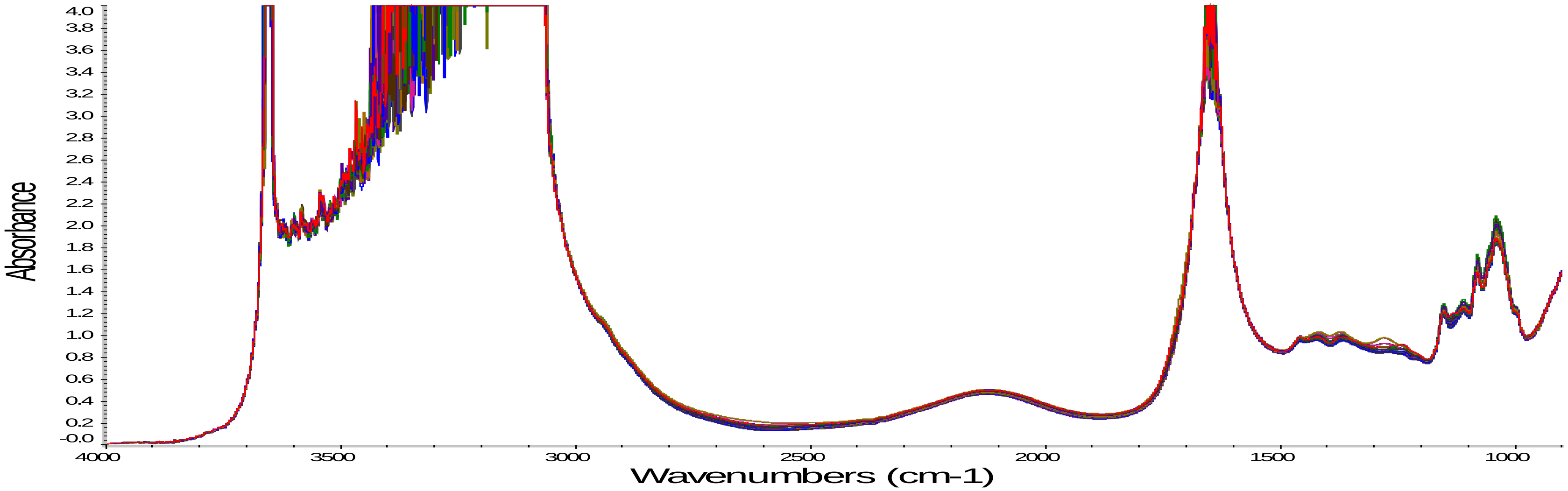


For SB2 matrix, linearity of 0.985 for LA and 0.979 for AA were achieved for the concentration range from blank to ~1.2%, which are quite similar to SB1 results shown in figure 2.

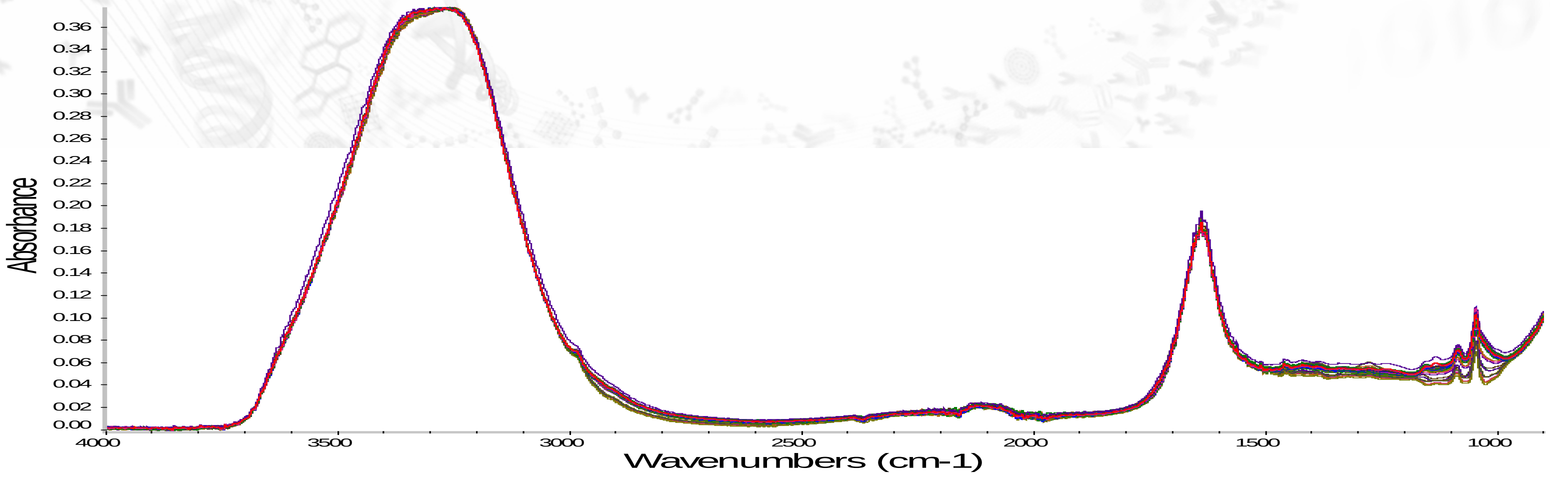

Quant Validation Plot for AA
R²=0.979
Concentration
Peak Area
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0

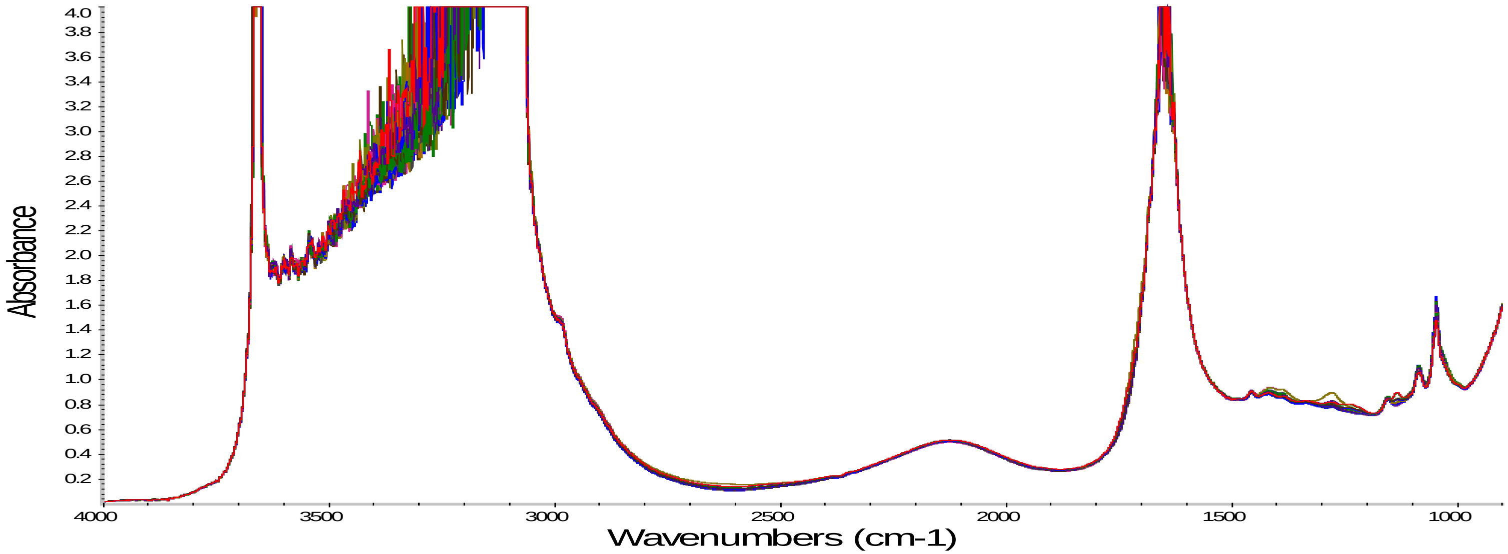


0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
Absorbance
4000
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)



4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
-0.0
Absorbance
4000
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)


Absorbance
Wavenumbers (cm-1)


Absorbance
Wavenumbers (cm-1)

Based on these results, it was demonstrated that using Dial path technology it is possible to quantitatively analyze acid components in these two matrixes and are certainly applicable to monitoring the fermentation process.