

# Integrated FT-IR Analysis System for Biodiesel

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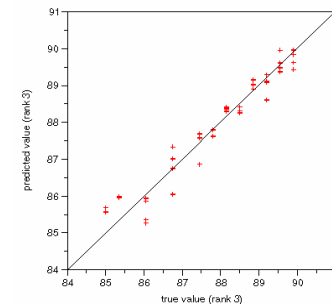
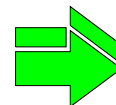
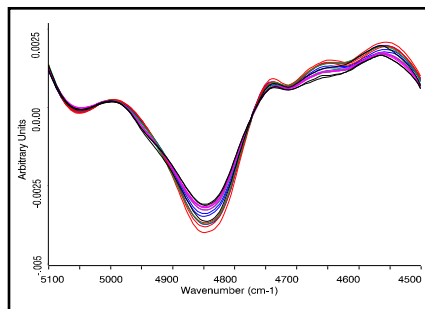
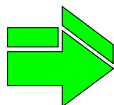
# FT-IR = Fourier Transform Infrared Spectroscopy

- Direct and indirect quantitative measurement of chemical and physical properties
- Fundamental absorption of molecular functional groups, 25-2.5  $\mu\text{m}$  (400-4000  $\text{cm}^{-1}$ )
- PLS chemometric algorithms used, not peak matching
- Results in less than two minutes
- No sample preparation!

# FT-IR Development & Operation

## Creating Calibrations

Component	A	B	C
Units	%	%	%
spectrum1	71.30	7.03	21.67
spectrum2	79.30	3.06	17.64
spectrum3	78.40	8.34	13.26
spectrum4	84.03	4.32	11.65
...	...	...	...
spectrum49	85.02	1.34	13.64
spectrum50	78.34	3.85	17.81

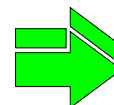
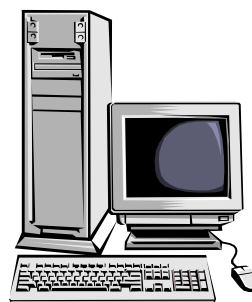
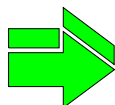
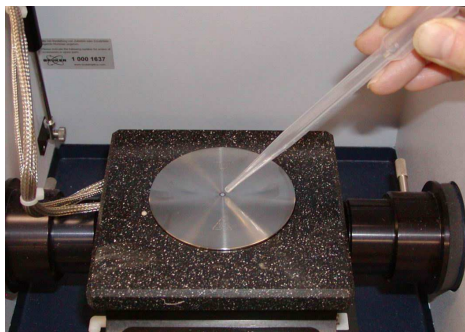


1. Prepare Standards

2. Collect Spectrum

3. Build , Optimize & Validate Model

## Analyzing Samples



Report	
Sample #081897-049	
Component A	81.55%
Component B	5.38%
Component C	13.06%

1. Measure Unknown

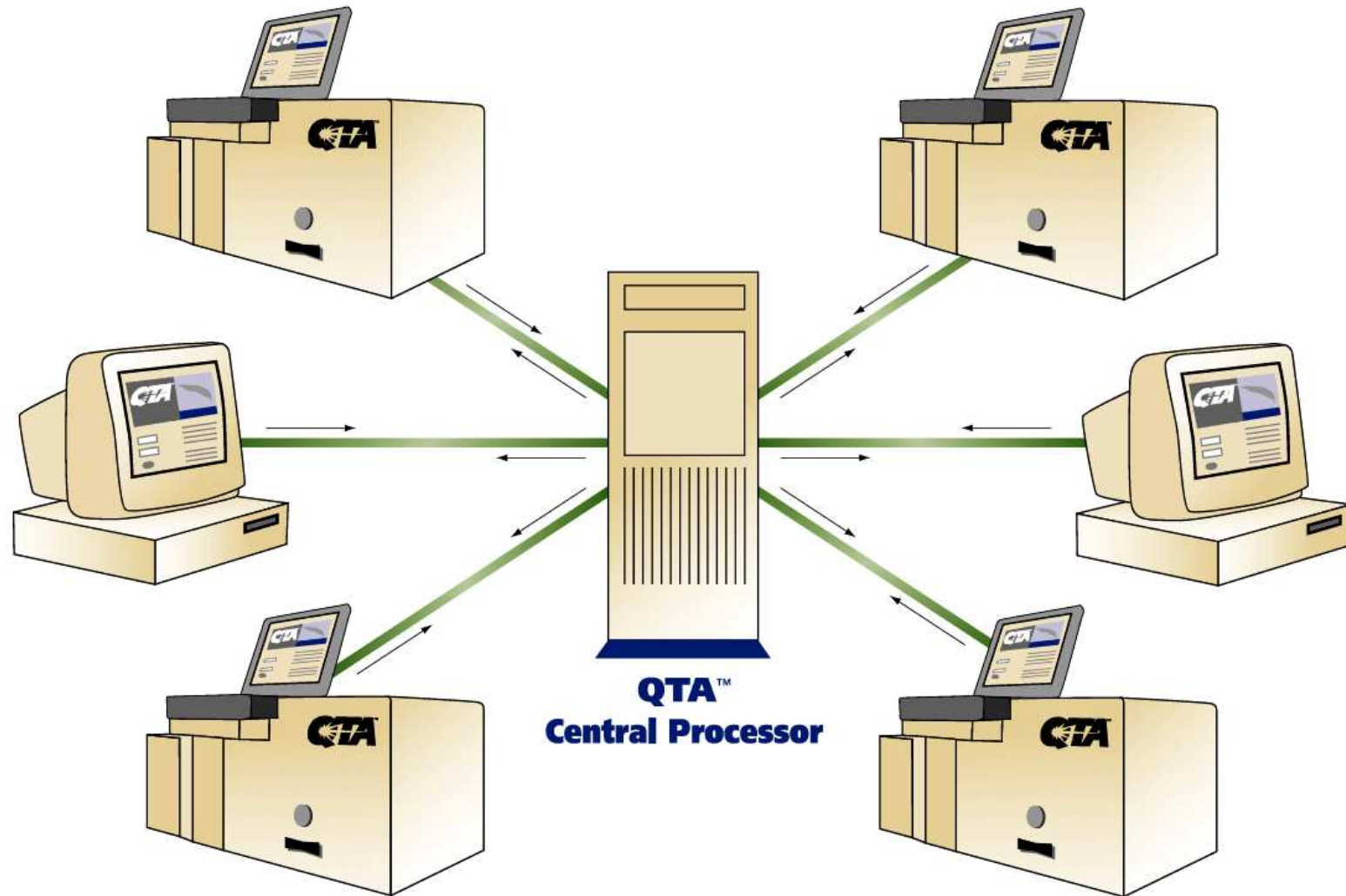
2. Access Model

3. Predict Concentrations

## Integrated Analysis System

- All FT-IR systems use a single central calibration
  - Minimum site to site variability
  - No on-site calibration adjustments
- Calibrations built and maintained by dedicated Ph.D chemometricians & spectroscopists
- Calibrations are based on primary data generated using ASTM methods from a certified lab
- Calibrations contain BD produced using 8 different RMs, and various blends

# Integrated FT-IR for Biodiesel Analysis



## Integrated Analysis System

- Calibrations contain as many as several hundred data points, and have been validated using hundreds more
  - Range and matrix effects are both incorporated
- New locations must undergo a validation process
- All instruments are monitored real time for instrument, system or operator error -- and no results returned if an issue exists
- Over 10,000 analyses per month using centralized calibrations

## Calibration statistics summary

	Correlation, R2, %		Std error of prediction	
	Global	Local	Global	Local
Water by KF, %	88	90	0.008	0.006
Acid number	88	72	0.13	0.10
Cloud point, deg C	93	--	1.4	--
Total glycerin, %	93	91	0.04	0.02
Free glycerin, %	96	94	0.009	0.002
Methanol, %	98	95	0.045	0.028
Mono-glycerides, %	94	--	0.12	--
Di-glycerides, %	98	88	0.07	0.05
Tri-glycerides, %	98	--	0.16	

# IR Round Robin for Biodiesel

- Samples from 6 different producers
- Multiple feedstocks
- Multiple production processes, batch vs. continuous
- Up to 9 IR instruments at various sites
  - 10 to 20 analyses per sample
- Up to 4 different wet chem labs
  - 2 - 6 analyses per sample
- For both QTA systems and wet labs:
  - same day blind replicates
  - different day blind replicates

## IR Round Robin for Biodiesel

- Average and standard deviation calculated for wet lab data for each sample
- Average and standard deviation calculated for IR data for each sample
- Bias calculated for each sample: average of wet lab – average of IR
- Average bias per trait calculated
- Average of sample std deviations calculated per trait for both wet lab and IR
- T-test calculated for each trait

## IR Round Robin for Biodiesel

	Both in spec	Wet lab in spec; QTA out of spec	Wet lab out of spec; QTA in spec
Total glycerin	28	1 (WL average = 0.21 %)	0
Free glycerin	28	0	1 (QTA = 0.019 %)

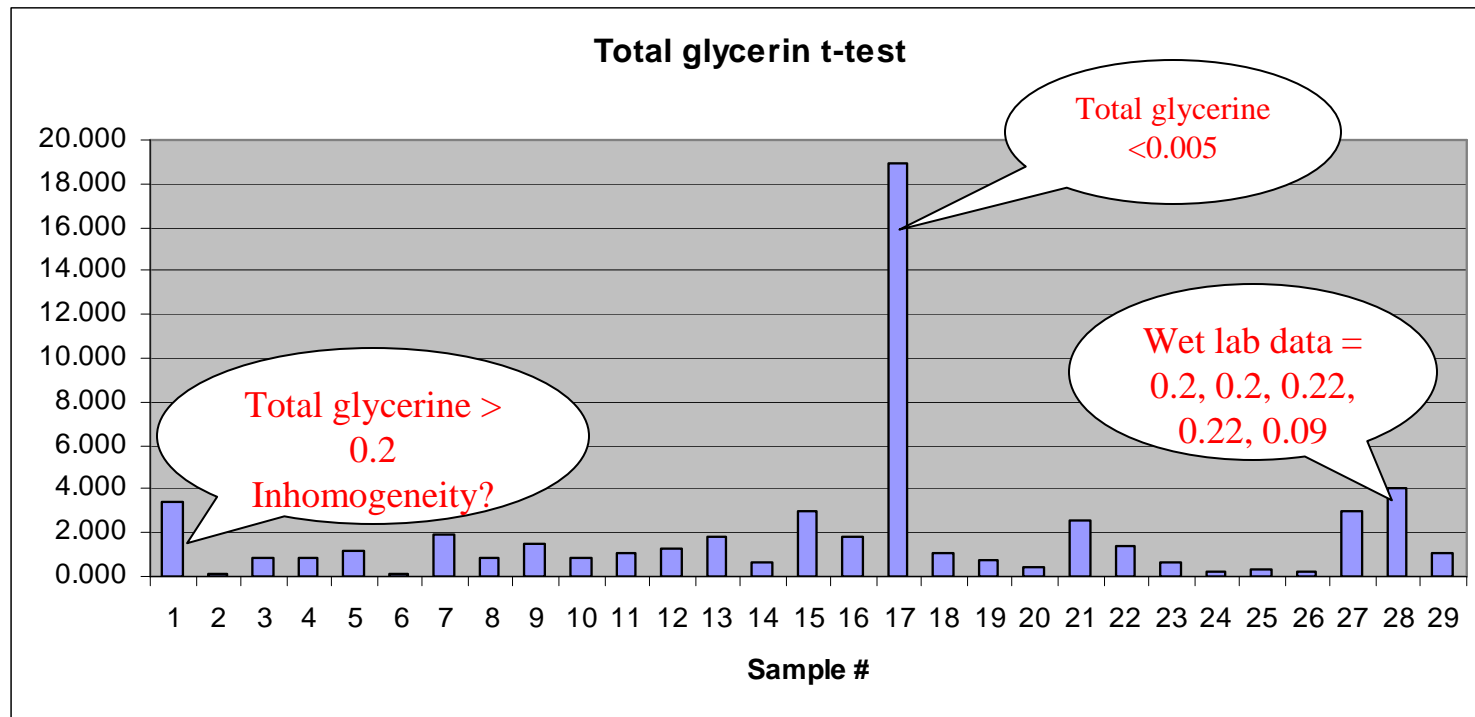
## IR Round Robin for Biodiesel

Property	Average Standard Deviation	
	IR	Wet lab
Moisture (KF), %	0.008	0.007
Acid Value	0.10	0.07
Cloud Point	0.85	2.30
Total glycerin, %	0.021	0.022
Free glycerin, %	0.0023	0.0059

## IR Round Robin for Biodiesel

Property	Bias	
	Average of wet labs - average of QTAs	t
Acid Value	0.03	1.8752
Cloud Point	-0.06	-0.6324
Total glycerin	-0.01	-3.2433
Free glycerin	0.001	-0.2464

# IR Round Robin for Biodiesel: t-test



# Integrated FT-IR for Biodiesel Quality Analysis

- High quality, accurate, robust calibrations are in place for BD quality analysis
- Variability of integrated FT-IR method is less than or equal to ASTM methods if calibrations are properly developed, validated & maintained
- Ease of use is significantly improved over ASTM methods

# Acknowledgements

- QTA Team
- Dr. Tom Ridgway, University of Cincinnati
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- All of the QTA customers who provided samples and analyzed samples for the RR testing