

The QTA[®] System for Biodiesel Analysis:

Pursuing approval as an alternate method for D6751

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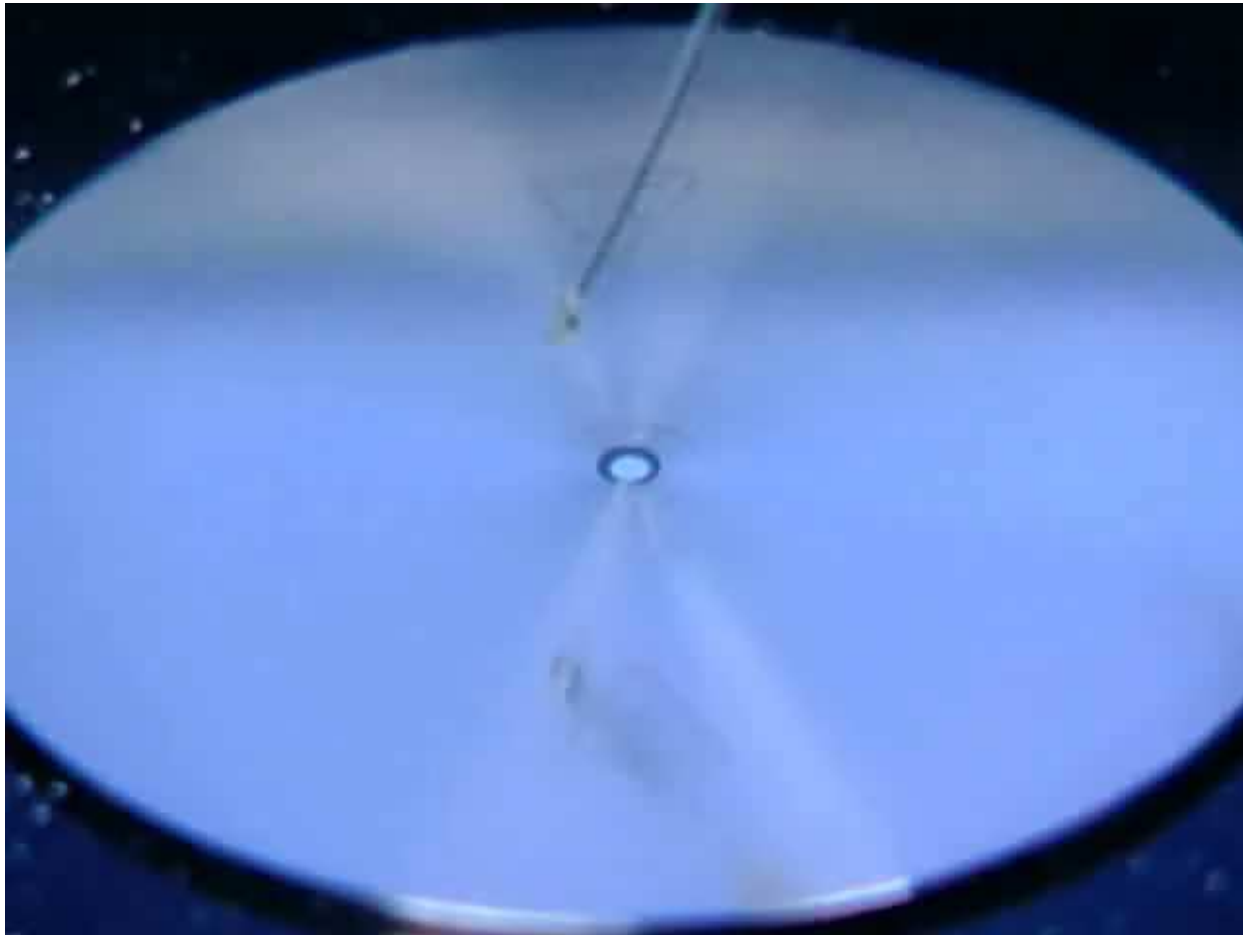
What is the QTA System?

- Fourier Transform Mid-Infrared Spectroscopy
- Networked Instruments
 - Single drop of liquid
 - No sample preparation
 - Light spectra of sample collected
 - Light spectra interpreted using mathematical equations
 - Results displayed



A faster, easier and accurate method for biodiesel analysis

Sample Video



Why use the QTA System for Biodiesel analysis?

- No sample preparation for B100 analysis
 - Minimizes error
 - Eliminates solvents & reagents
 - Reduces waste
- Easy to Use
 - All calibration and standards done by Cognis
 - No sample prep
 - Enter sample ID, hit Analyze
- Versatile
 - Provides results for multiple properties in a single test
 - Analyzes B100, in process samples, glycerin, methanol, and oil



Producers & users of biodiesel can analyze more frequently to ensure consistently high quality – over 1.5 million analyses were conducted using the QTA System in 2008 and 2009

The QTA System's capabilities for B100 are broad:

	Commercially available	Under development
B100	<ul style="list-style-type: none"> • Moisture • Cloud Point • Acid Number • Total and Free Glycerin • Mono-, Di- and Tri-glycerides • Ester content • Iodine Value • CFPP • Viscosity • Density 	<ul style="list-style-type: none"> • Oxidative Stability • Cold Soak Filtration • Mono-palmitin • Mono-stearin • Mono-olein

Unlimited Analysis Packages start at \$3500 per month
Per Sample Analysis Packages as low as \$10 per sample

The QTA System can also analyze other materials

	Commercially available	Under development
In process biodiesel	Mono-, Di- and Tri-glycerides Bound Glycerin	
Oils & Fats	Moisture, FFA	
Glycerin	Moisture, Ash, Methanol, % glycerin	
Biodiesel blends	% biodiesel in diesel	D7467 properties

Why does the QTA system work?

- Calibrations developed and validated using thousands of real-world samples
 - Calibrated using a wide variety of feedstocks: soy, canola, WVO, poultry, tallow, CWG, sunflower, and mixtures thereof; also jatropha, fish oil and algae
 - And the feedstock does not need to be identified for the analysis
 - Biodiesel samples from U.S., Canada, Europe, South America
 - Samples from production facilities, terminals, storage tanks, and pilot facilities
 - Samples included from 2005 through 2010
- Networked system ensures that all use the same calibrations
 - Minimizes lab to lab variability
 - Accuracy is maintained by trained, dedicated chemists
- Ongoing validation of new facilities, new processes, new feedstocks

As a part of NBB's initiative for faster, easier test methods, the QTA method is being balloted for inclusion in ASTM D6751

- Round robin was conducted in 2008 using AOAC and ASTM guidelines to compare QTA methods with ASTM test methods
- QTA method became an AOCS Standard Practice, CK 2-09, in late 2009
- Based on the results of the round robin, CK 2-09 is proposed for inclusion in ASTM D6751 as an alternate method for:
 - Total and free glycerin
 - Cloud point
 - Methanol content
- Once included in D6751, QTA can be used under BQ-9000

Timing:

Subcommittee ballot in June 2010

Main committee ballot in December 2010



Round robin details

- Eight labs using primary test methods
 - Water by Karl Fisher: ASTM D6304
 - Acid Number: ASTM D664
 - Total and Free Glycerin: D6584
 - Methanol content: EN 14110
- Eight different labs using the QTA method
- Ten samples to each lab
 - Eight fuels + 2 blind duplicates, randomized
 - Five different feedstocks, three of them were blends of fats & oils
 - Seven different production facilities
 - Duplicate analyses on each sample at each lab



Round Robin results show good performance by QTA

Property	D6708 Result	Reproducibility at spec limit	
		ASTM / EN method	QTA
Water by KF	Equivalent under D6708	0.017	0.019
Cloud point	Equivalent under D6708	2.0	2.9
Methanol	Equivalent under D6708	0.116	0.1
Total Glycerin	Not equivalent under D6708, but better reproducibility	0.1	0.054
Free Glycerin	Not equivalent under D6708, but equivalent when 1 fuel removed	0.013	0.012
Acid Number	Not equivalent under D6708	0.15	0.218

Round robin demonstrated the QTA System's suitability as a faster, easier alternate method

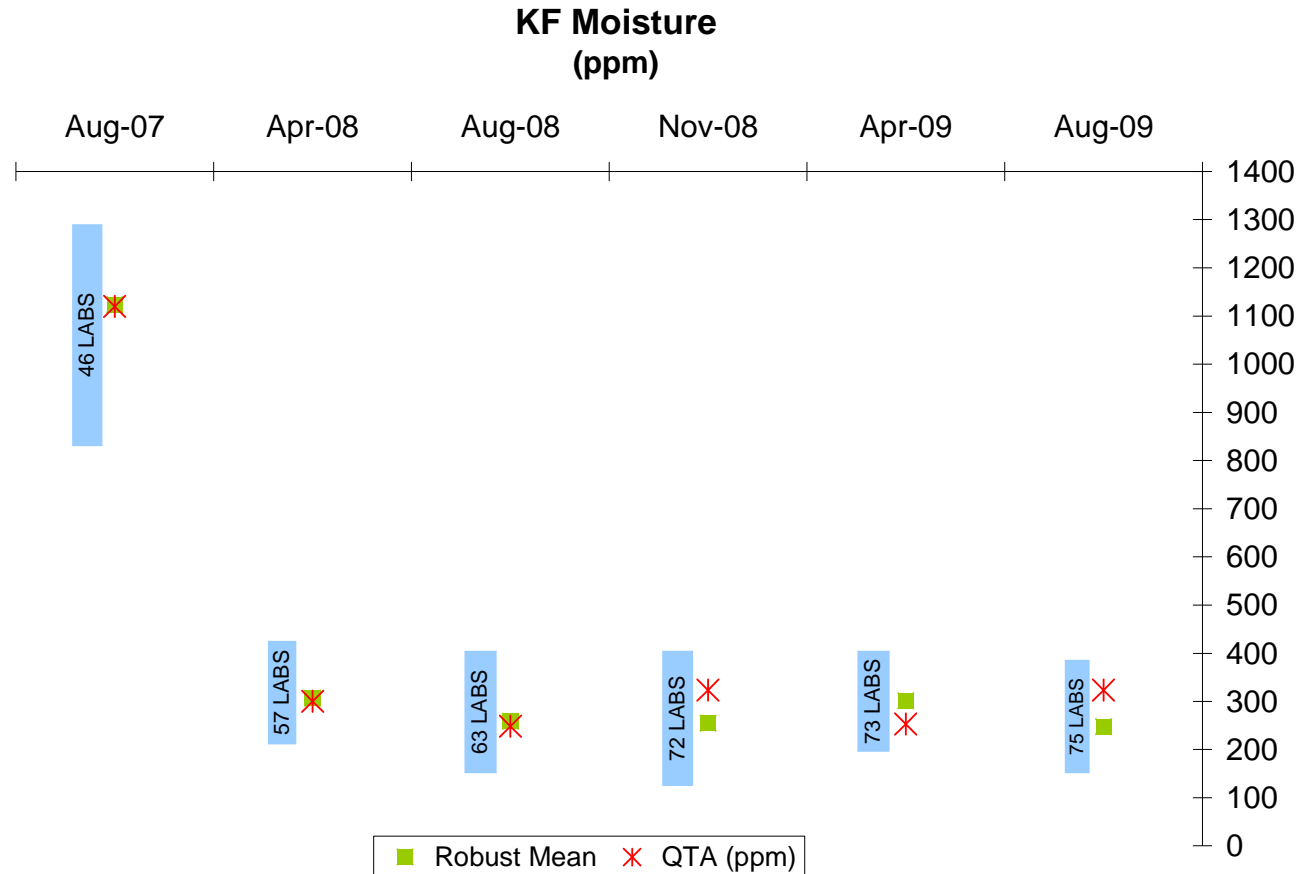
Summary of Round Robin results:

- Using D6708, QTA is statistically equivalent to the approved wet chemistry methods for Karl Fisher moisture, Methanol and Cloud Point
- For both total glycerin and free glycerin, QTA reproducibility is significantly better than D6584
 - TG reproducibility = 0.054% across the spec range
 - FG reproducibility ranges from 0.006 – 0.0124 across the spec range
 - QTA predictions for FG error on the high side, identifying sterol glucosides, another bad actor.
- More work is needed on acid number, with two fuels causing the methods not to be equivalent

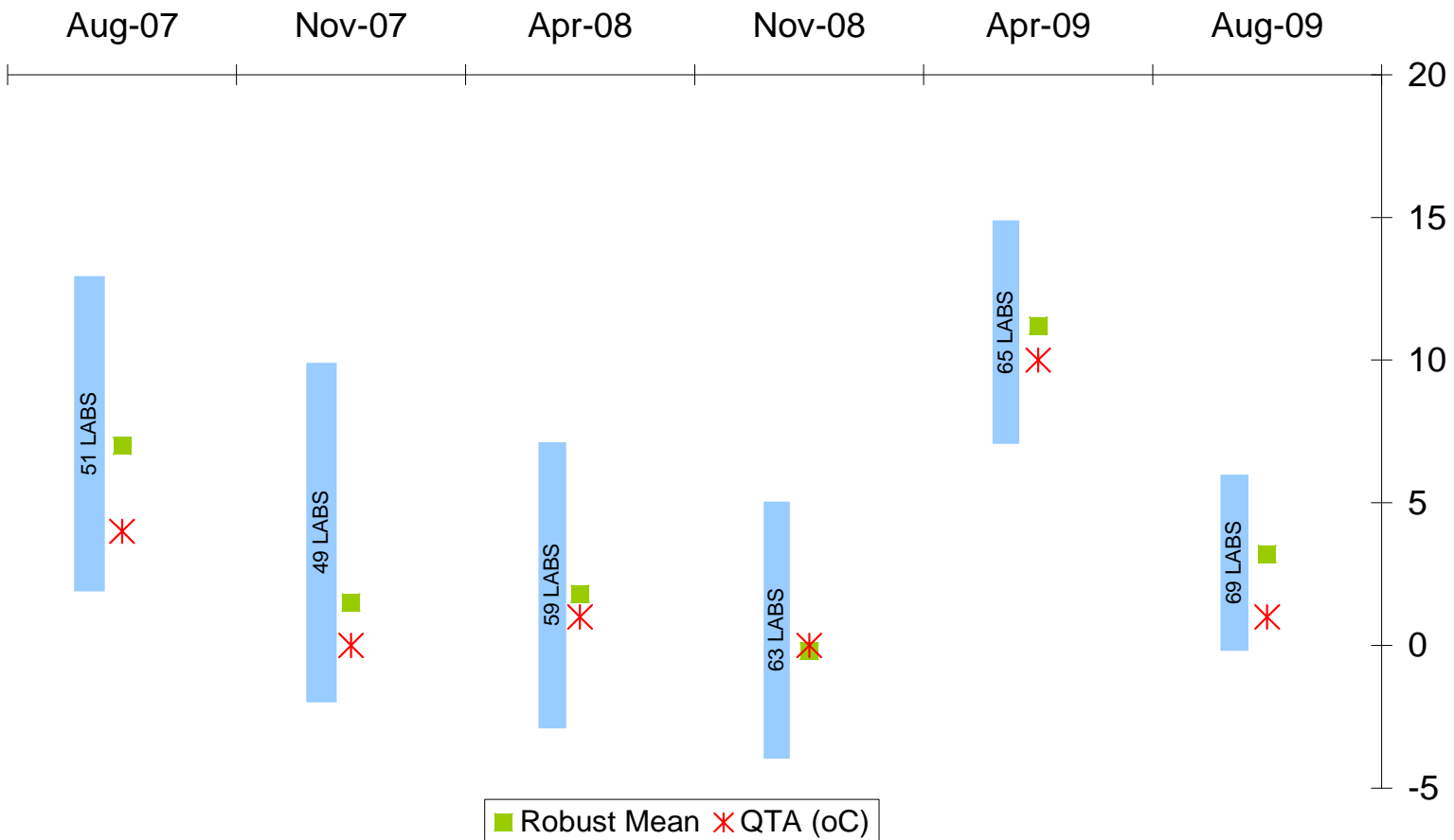
QTA also participates in the ASTM Interlaboratory Crosscheck Program

- As many as 70 labs participate
- No “right” answer is given, but may be inferred from the mean of the analysis results of the many labs
- The subsequent graphs show the following:
 - The blue bar is the range of results from the various laboratories
 - The green point is the robust mean of the results from the various laboratories
 - The red X is the QTA result

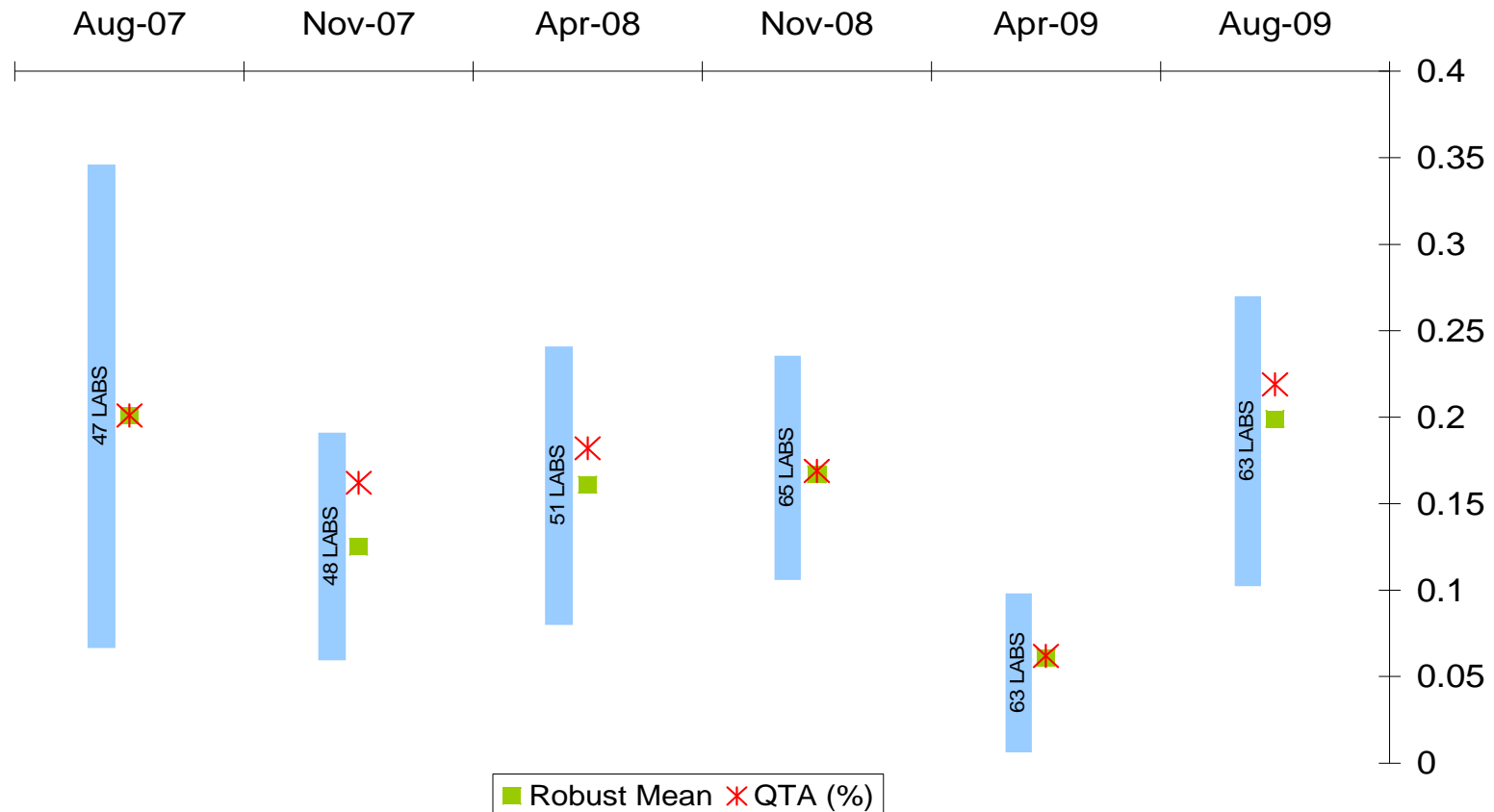
QTA shows excellent agreement with ASTM ILCP results for Moisture by Karl Fisher



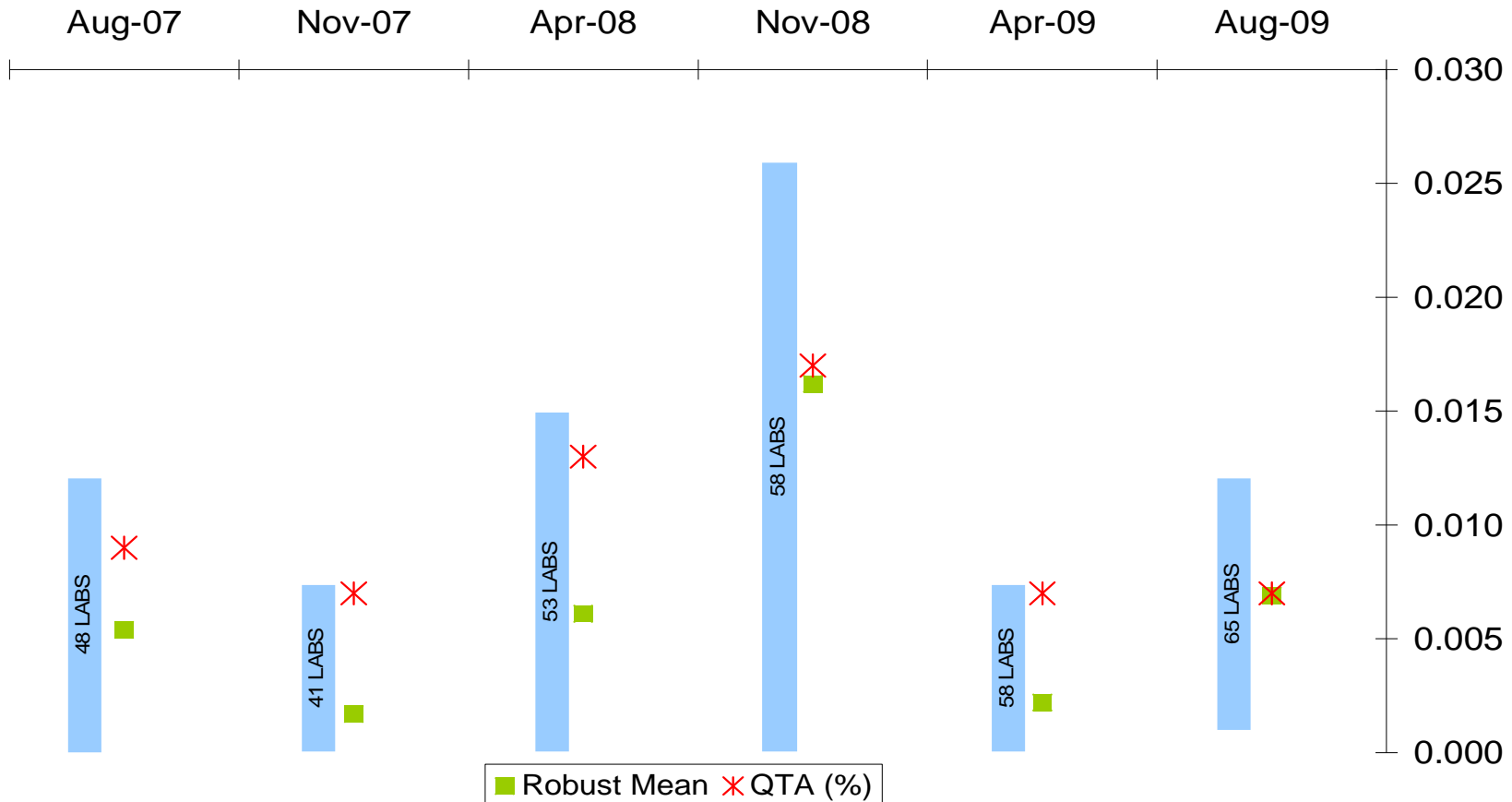
QTA shows very good agreement with ASTM ILCP results for Cloud point, deg C



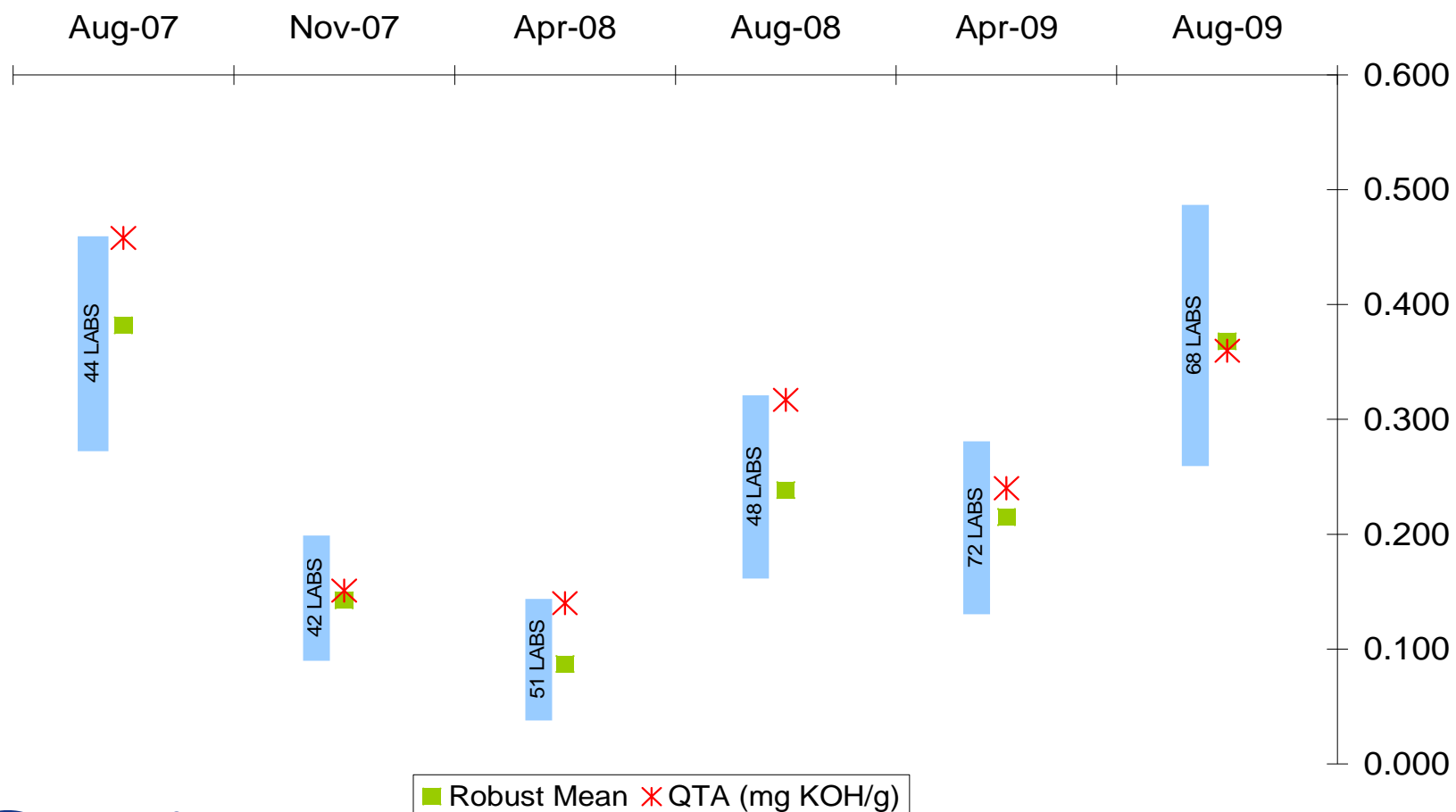
QTA shows excellent agreement with ASTM ILCP results for Total Glycerin



QTA shows good agreement with ASTM ILCP results for Free Glycerin



QTA shows good agreement with ASTM ILCP results for Acid Number



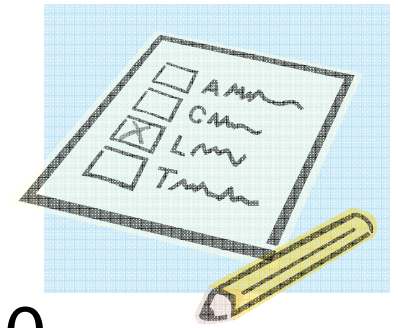
The QTA System performs well across the board in ASTM ILCP studies

- In all cases, the QTA system results fell within the range of results provided by the laboratories participating in the ILCP, and in most cases, predicted close to the robust mean
- ILCP results will continue to be used to monitor the ongoing performance of the QTA System

The QTA System is a viable alternate method for current ASTM methods for biodiesel analysis

- Both the round robin data and the ILCP data collected over the past 2 years demonstrate that the QTA system provides accurate results for key parameters
- The QTA System offer significant advantages in ease of use and eliminates the use of solvents and reagents
- Approval of the QTA System as an alternate method for analysis by biodiesel producers and users will encourage more frequent testing and better quality control

QTA System approval under ASTM D6751 is envisioned for 2010



- Subcommittee ballot in June 2010
- Main committee ballot in December 2010

*Once approved as an alternate method in D6751,
QTA can be used under BQ-9000 protocols*