



Results from the Cognis QTA Evaluation

Gina M. Clapper
AOCS Technical Services
&
Barbara Stefl
Cognis Corporation



Collaborative Study

Purpose

- ❖ Evaluate the results provided by the Cognis QTA system to primary methods

Participants

- ❖ Primary Method
 - 8 labs (1 Brazil and 7 USA)
- ❖ QTA
 - 8 user sites in the U.S.



Parameters and Methods

Water

ASTM D6304

Acid number

ASTM D664

Cloud point

ASTM D2500

Total glycerin

ASTM D6584

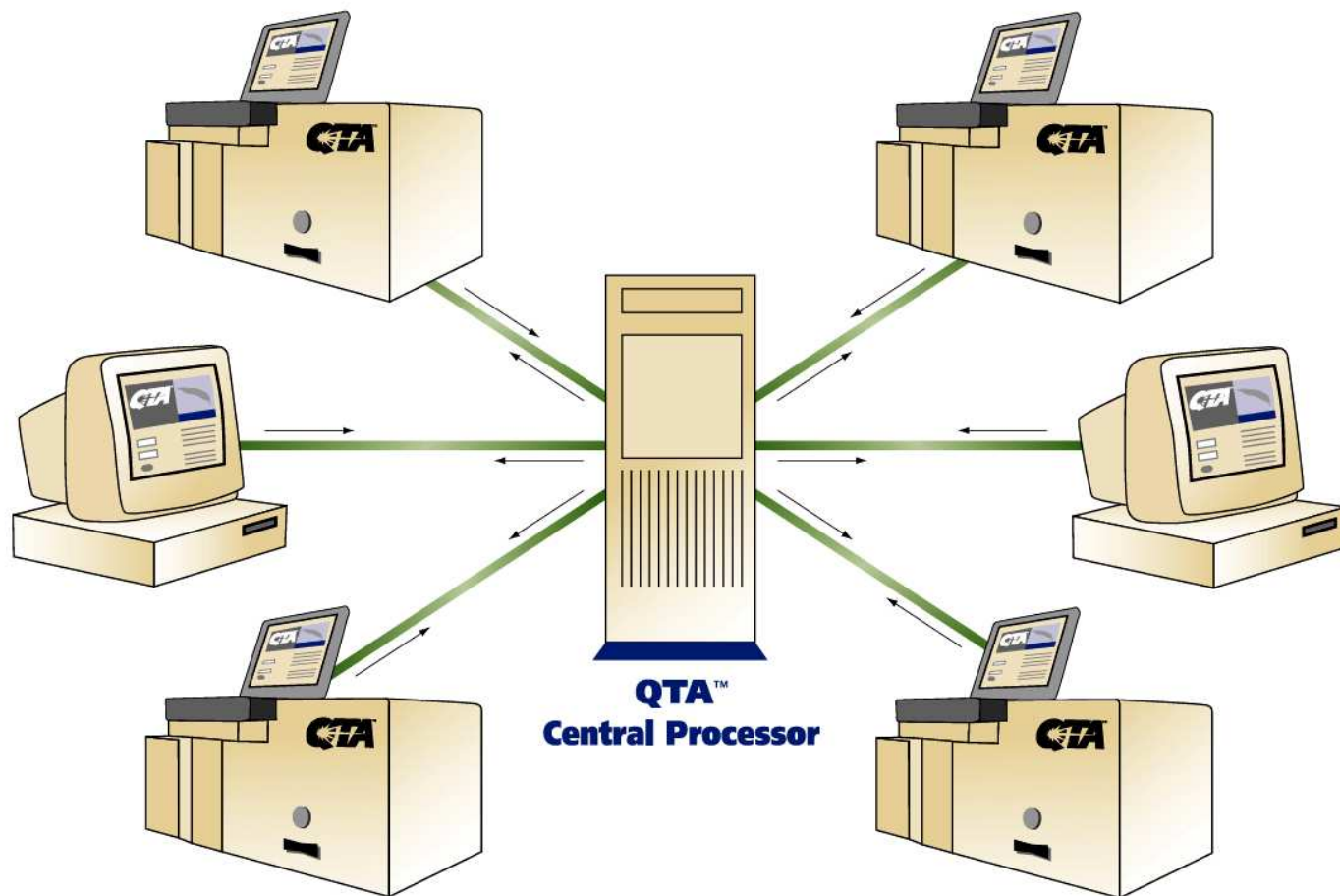
Free glycerin

ASTM D6584

Methanol

EN14110

The QTA® System: A networked FT-IR system





The QTA® System: A networked FT-IR system

- Calibrations are centrally built and centrally maintained
- All QTA users use the same calibration
- Calibrations are validated for each user and updated periodically for “good outliers”
- Primary data is generated using ASTM methods predominantly using a single laboratory, although multi-lab data has been thoroughly evaluated



The QTA® System: A networked FT-IR system

- No sample preparation is required to analyze biodiesel using QTA
- Analysis of all six parameters can be conducted in less than 2 minutes
- Use of QTA encourages more frequent biodiesel testing since it is quick and easy



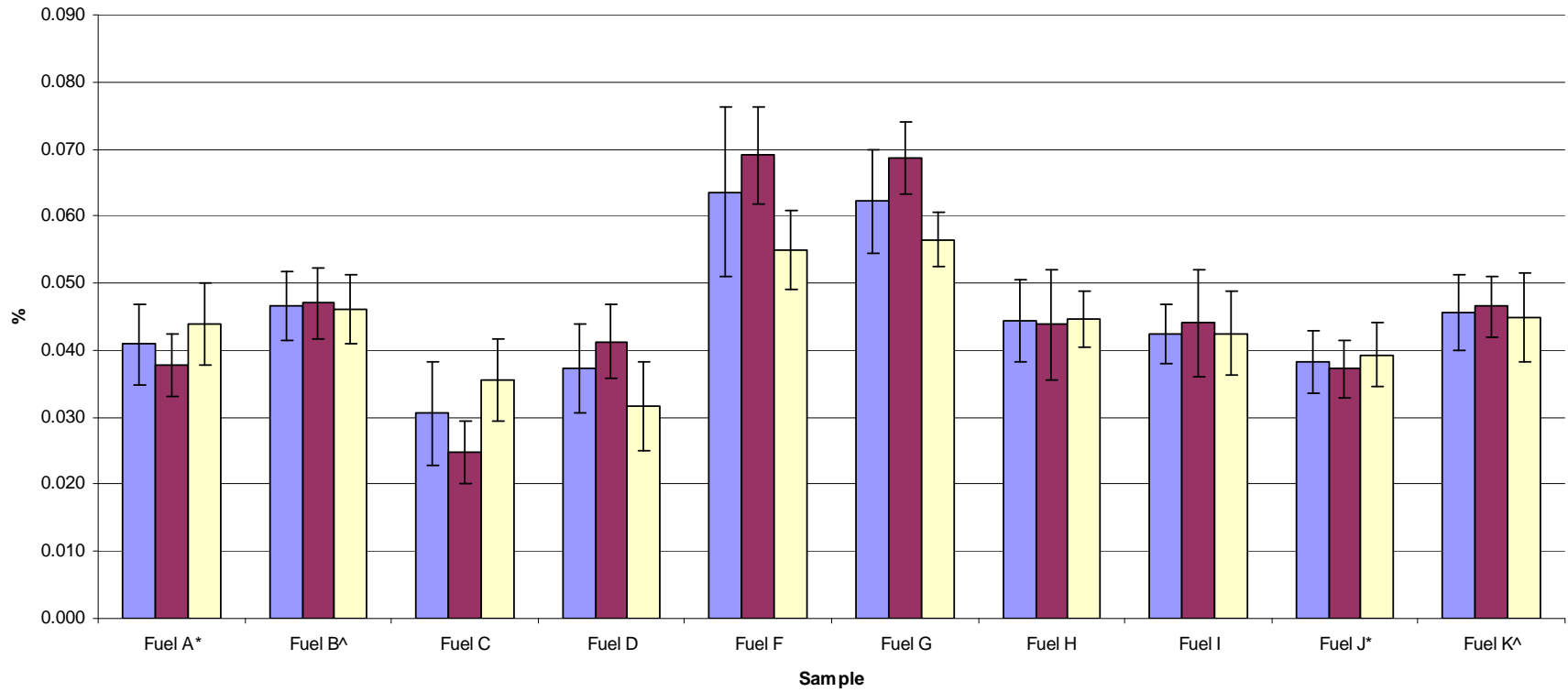
Results

- Sorted by “Fuel” source
- Analyzed by the AOAC International Interlaboratory Statistical Program for Blind Duplicates 2001, version 1.7
- Analyzed by D6708



Karl Fisher Moisture

■ Total ■ Primary □ QTA





D6708 Results for KF water

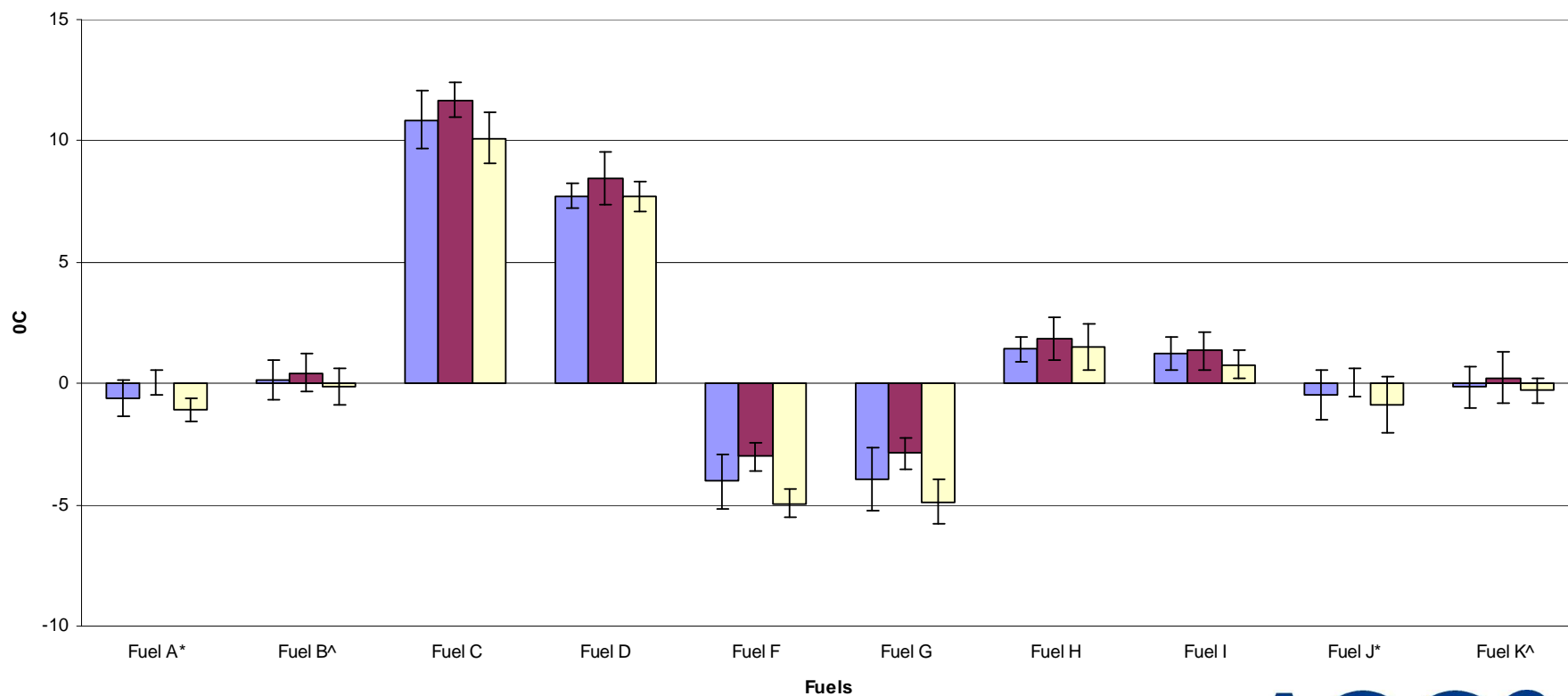
At spec limit	ASTM/EN method	QTA
Repeatability	0.0014	0.012
Reproducibility	0.01	0.019

 D6708 result = PASS



Cloud Point

■ Total ■ Primary □ QTA





D6708 Results for cloud point

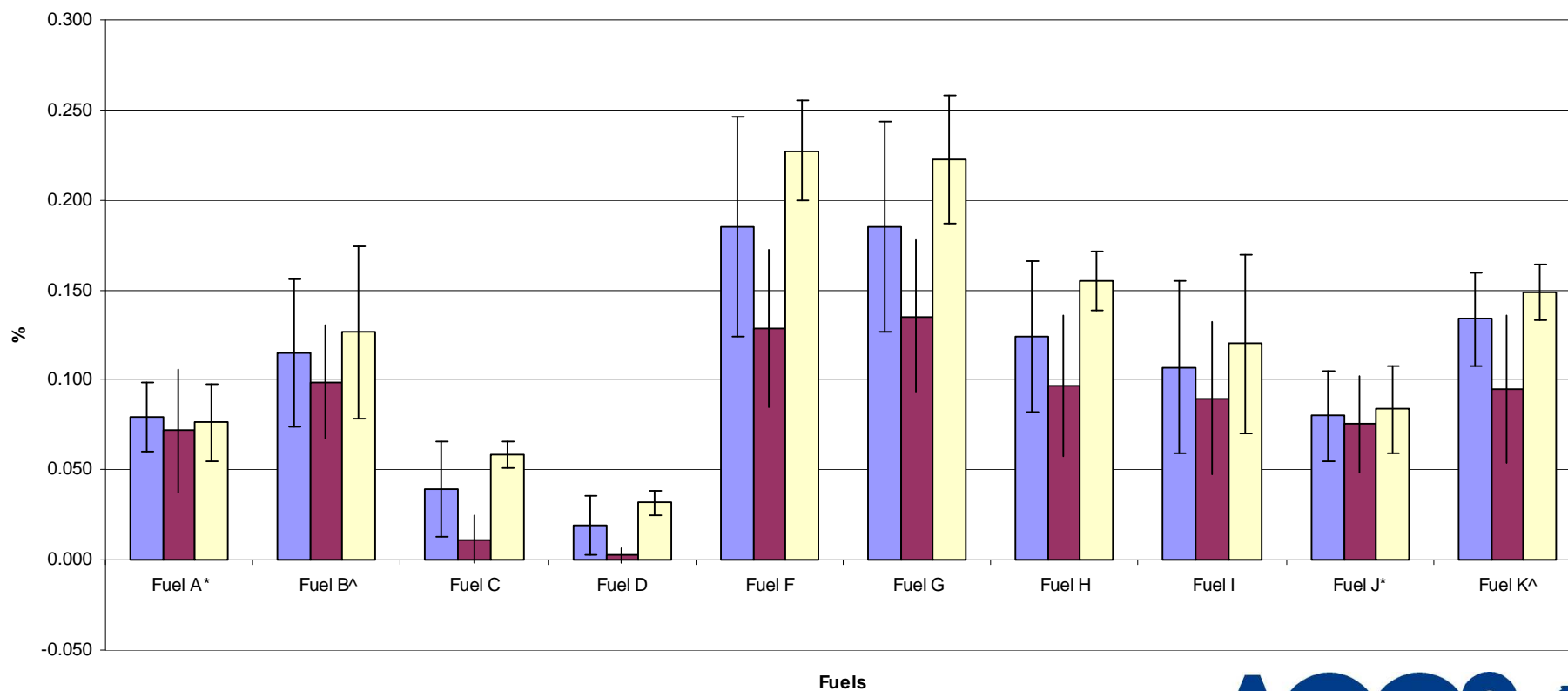
At spec limit	ASTM/EN method	QTA
Repeatability	0.636	1.28
Reproducibility	1.965	2.89

 D6708 result = PASS



Methanol Content

■ Total ■ Primary □ QTA





D6708 Results for methanol

At spec limit	ASTM/EN method	QTA
Repeatability	0.017	0.07
Reproducibility	0.116	0.10

 D6708 result = PASS



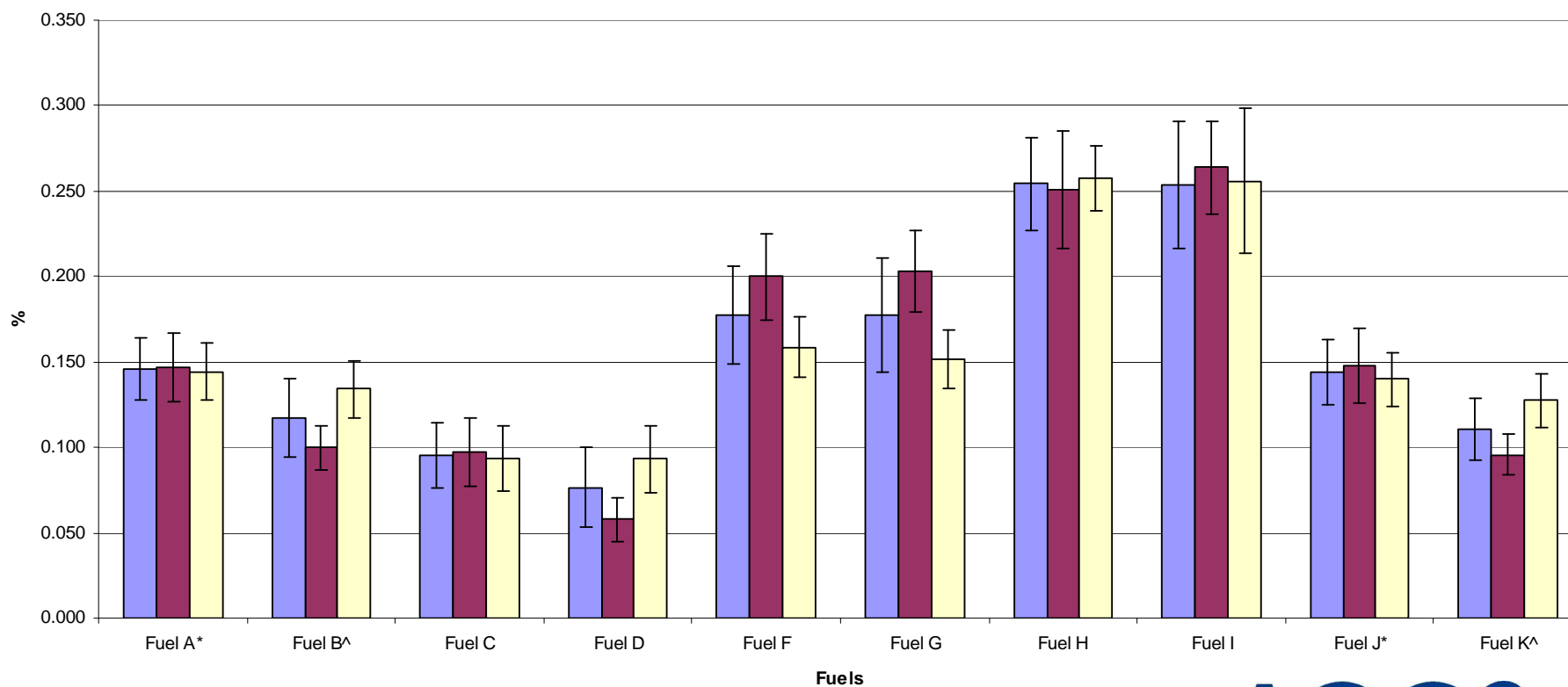
Summary for water, cloud point, methanol

- QTA passes D6708 for all three
- Repeatability of QTA about double that of ASTM methods
- Reproducibility:
 - ❖ KF = about the same
 - ❖ Cloud point = QTA not as good by 0.7 deg
 - ❖ Methanol = QTA twice as good



Total Glycerin

■ Total ■ Primary □ QTA



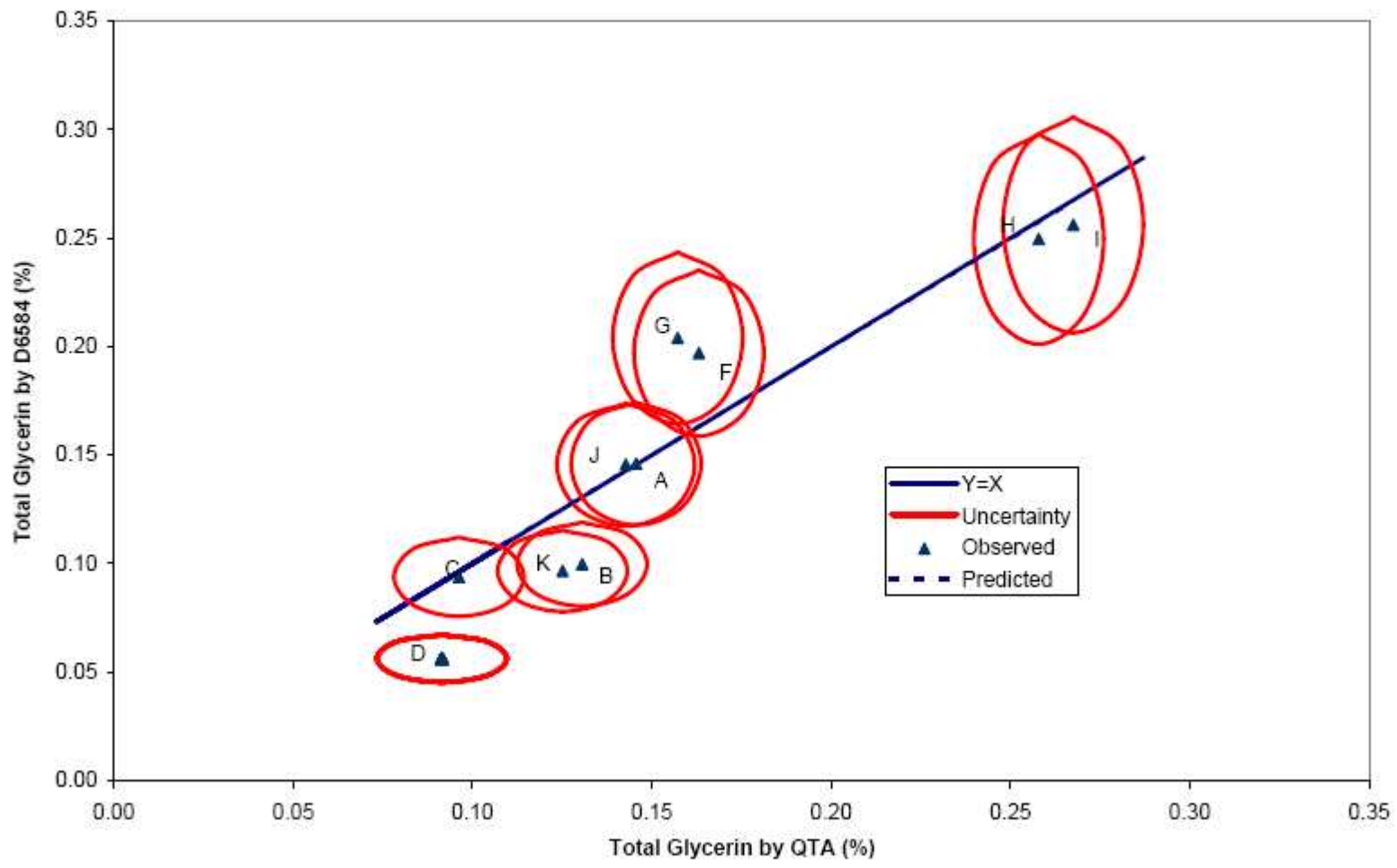


D6708 Results for total glycerin

At spec limit	ASTM/EN method	QTA
Repeatability	0.01	0.035
Reproducibility	0.1195	0.054

🌐 D6708 result = FAIL

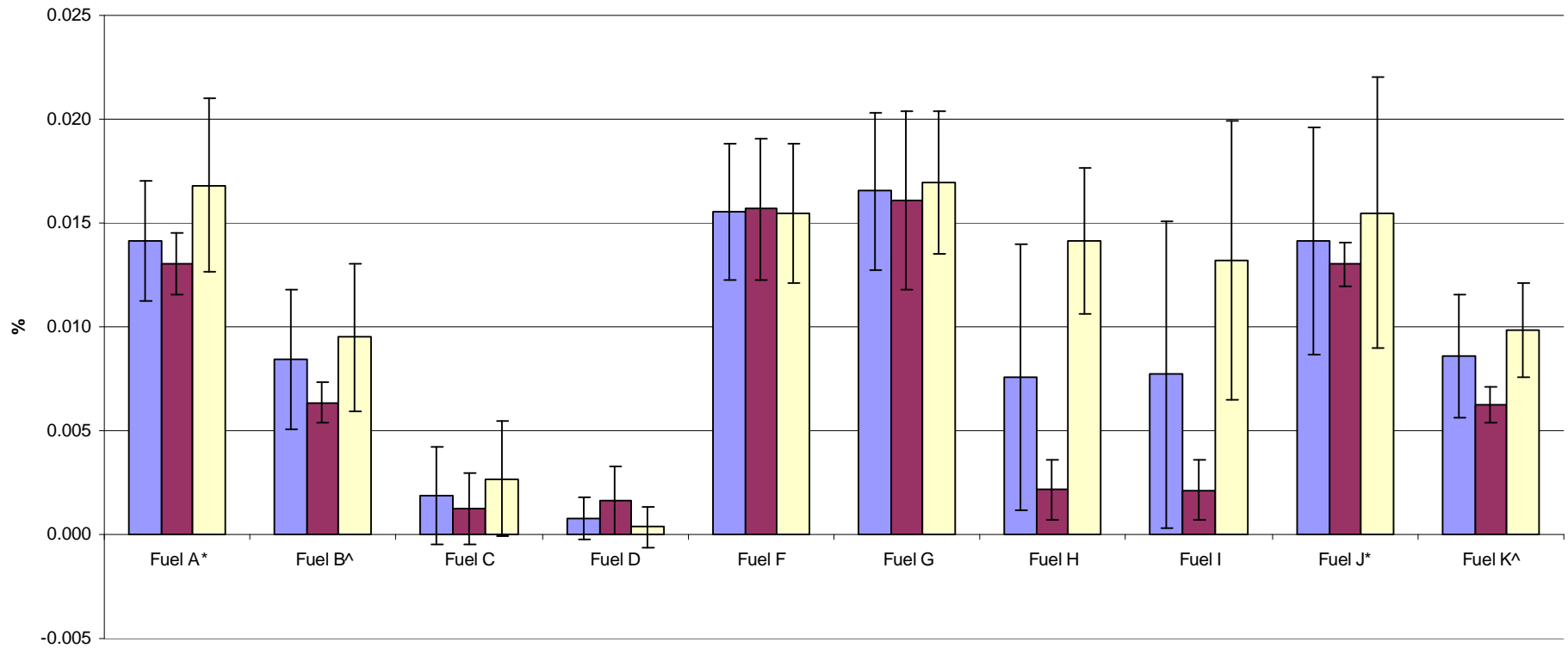
Confidence Plot





Free Glycerin

■ Total ■ Primary □ QTA



Fuels

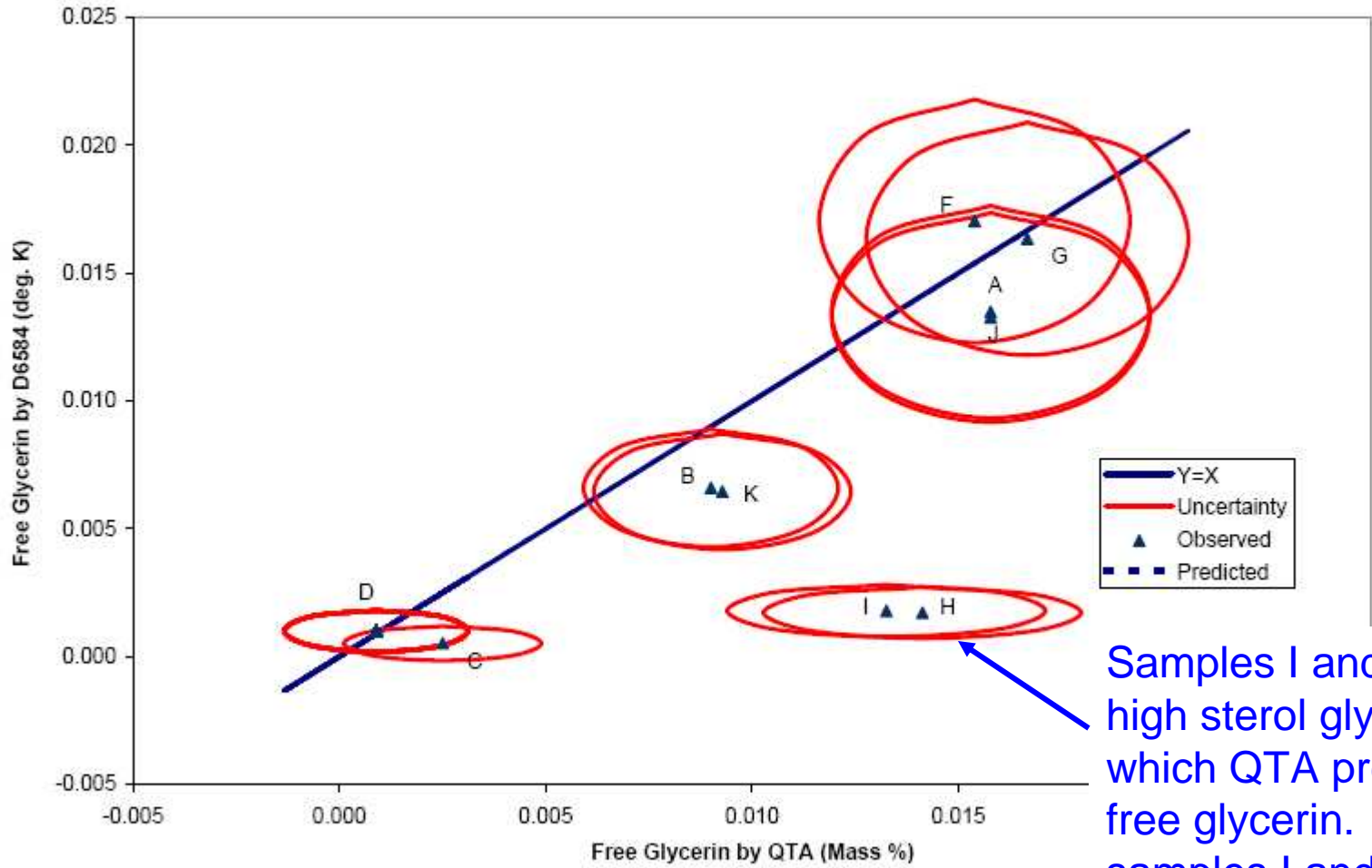


D6708 Results for free glycerin

At spec limit	ASTM/EN method	QTA
Repeatability	0.004	0.0075
Reproducibility	0.0135	0.006

🌐 D6708 result = FAIL

Confidence Plot



Samples I and H have high sterol glycosides which QTA predicts as free glycerin. If samples I and H are removed, QTA passes D6708



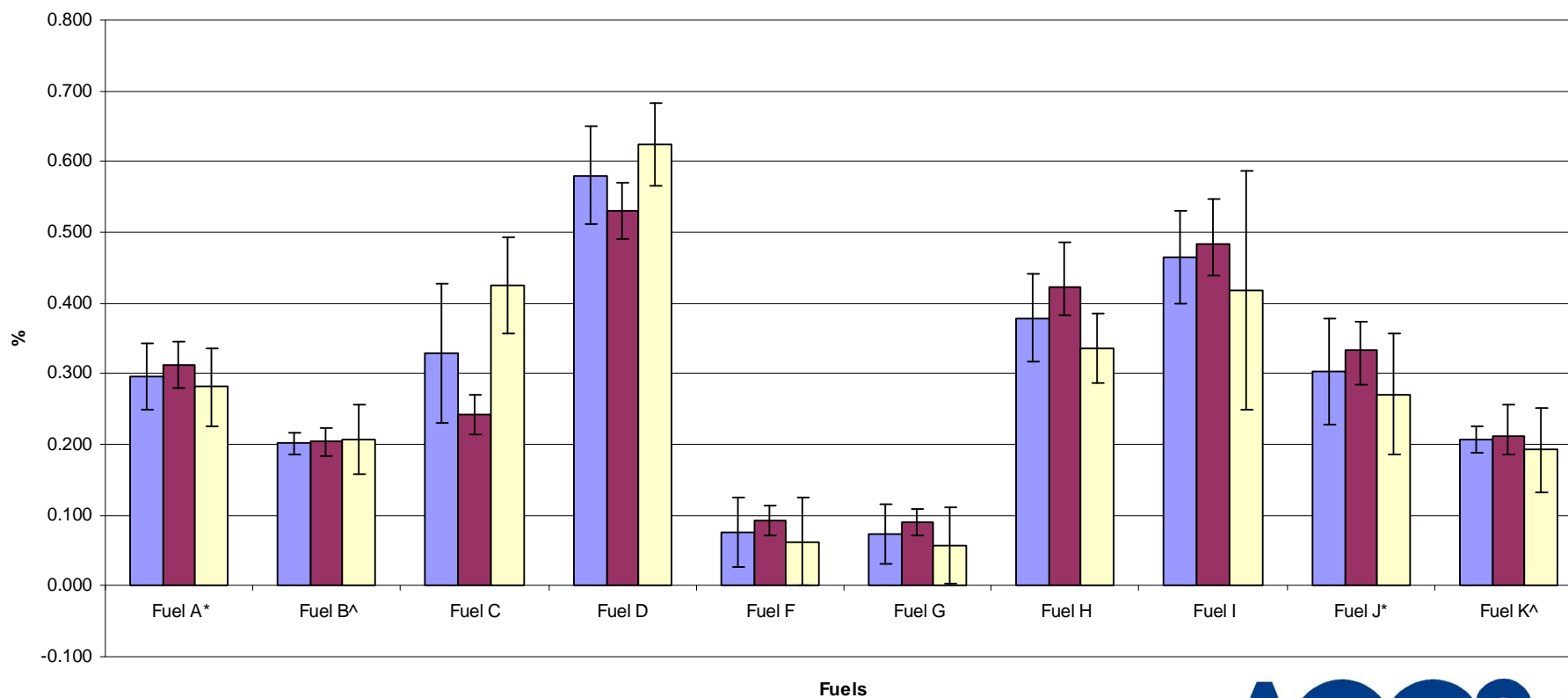
Summary of D6708 results for TG and FG

- D6584 for TG is not capable at the specification limit, with a reproducibility of 0.12 or half of the spec limit
- QTA fails equivalency test for TG, but is significantly more capable
- Reproducibility for both TG and FG using QTA is significantly better than D6584
- When 2 samples with sterol glucosides are removed, QTA passes D6708 for FG



Acid Value

■ Total ■ Primary □ QTA



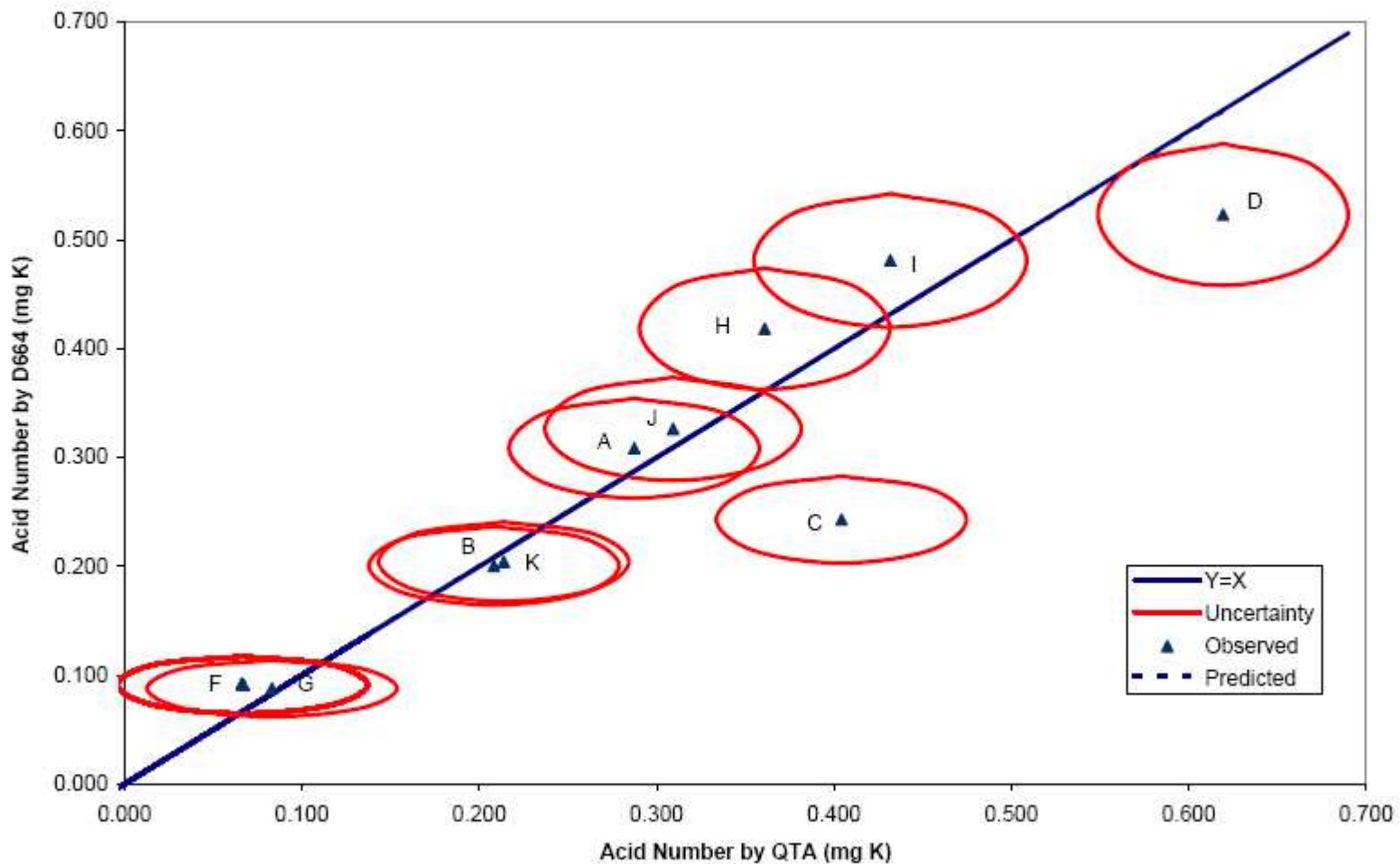


D6708 Results for acid value

At spec limit	ASTM/EN method	QTA
Repeatability	0.026	0.172
Reproducibility	0.154	0.219

🌐 D6708 result = FAIL

Confidence Plot





Discussion

- Using D6708, QTA is statistically equivalent to the approved wet chemistry methods for KF, Methanol and Cloud Point
- D6584 is not capable for total glycerin, with the reproducibility at the spec limit of 50% of the range. QTA reproducibility is significantly better at 0.054% across the spec range
- For FG, QTA predictions error on the high side, identifying SG, another bad actor. Reproducibility is significantly better than D6584.
- More work is needed on acid number, with two fuels causing failure vs. D6708



Next steps

- QTA test method has been submitted to AOCS committee on Rapid and Non-Destructive Testing for balloting as an official AOCS Standard Procedure
- Once an AOCS method number is received for the QTA method, it is proposed that the method be included as an alternate method within D6751 for total & free glycerin, cloud point and methanol
- Additional work and round robin will be conducted on acid value and any future methods to secure D6708 "Pass"