Biodiesel Quality

Test Methods Update

Quality Update

CGSB and Biodiesel Test Methods

- Can/CGSB-3.520 Automotive Low Sulphur Diesel Fuel Containing Low Levels of Biodiesel Fuels (B1 – B5) is published
- same technical requirements as 3.517, the LSD standard.
- This specification allows biodiesel blends up to 5% to be incorporated into Low Sulphur Diesel (LSD)

CGSB and Biodiesel test Methods – Next Steps

- CGSB is currently balloting two amendments to 3.520: Amendment 1 would add ULSD to the standard; Amendment 2 included some rewording to align 3.520 with 3.522
- CGSB is currently balloting 3.522, the proposed B6-B20 standard which would be restricted to ‘knowledgeable buyers’ and not for sale to the general public.

ASTM and Biodiesel Test Methods

- D 6751 Ballot Items
- Biodiesel Stability Working Group Update

D 6751 Scope Change

- Allow biodiesel to bring cetane and lubricity into specification (sulfur and aromatics already allowed)
- The Biodiesel Task Force came to consensus on these changes as they could potentially enlarge the overall diesel fuel pool while providing needed cetane and lubricity requirements for the finished fuels.

Acid number from 0.8 to 0.5

- About 30% of North American doesn't meet 0.5
- Acid number change negotiated between OEM's and biodiesel suppliers and agreed to
- Needed to secure better OEM support for B20
- Large body of experience with 0.5 in Europe
- Acid number is known to directly correlate with oxidative stability.
- Lowering acid number will help stability
- Harmonized acid number with the European specification EN14214

Viscosity from 6.0 to 5.0

- This will help ensure quality
- The results of a B100 fuel survey of 27 samples, indicate none of biodiesel samples fell beyond the 5.0 maximum proposed limit.
- Harmonizes viscosity with European specification EN14214

Ca/Mg combined 5 ppm max

- Some engine and fuel injection equipment manufacturers have requested a specific limit on Ca/Mg due to concerns with injector coking and adverse effects on new particulate trap technologies
- The results of a B100 fuel survey of 27 samples, indicate approximately 30% of biodiesel samples fell between the proposed maximum levels of 5 ppm and 10 ppm
- Harmonizes with European specification EN14214

Na/K combined 5 ppm max

- Some OEM and FIE manufacturers have requested a limit on Na/K due to concerns with soap formation and adverse effects on particulate trap technologies
- These compounds are common catalysts for the biodiesel reaction and can be present if not properly removed during processing
- The results of a B100 fuel survey of 27 samples, indicated **no** biodiesel outside the proposed maximum levels of 5 ppm.
- Harmonizes with European spec EN14214

Replace D 2709

- Replacing D 2709, Water and Sediment, with D 6304 Karl Fisher Moisture and D 6217 Particulate Contamination Filtration methods.
- Moisture specification limit by D 6304 of 0.050 vol%
- Particulate contamination specification limit of 24 mg/kg
- Harmonizes with European specification EN14214

ASTM Oxidation Stability Working Group (WG) Update

- Significant increase in understanding the factors that influence the aging phenomenon with biodiesel and biodiesel blends
- Major factors influencing stability are:
  - Presence of partially reacted or un-reacted oils
  - Level and type of un-saturation, 18:2 and 18:3
  - Presence of anti-oxidants, either natural or added

Biodiesel Stability Issue

- ASTM D6751 should include a stability parameter

- B20 Operational problems are few, and when they occur are usually caused by biodiesel not meeting existing requirements of D6751 (high glyceride or soap content) or other issues (cleaning effect, cold flow, microbial, improper blending)

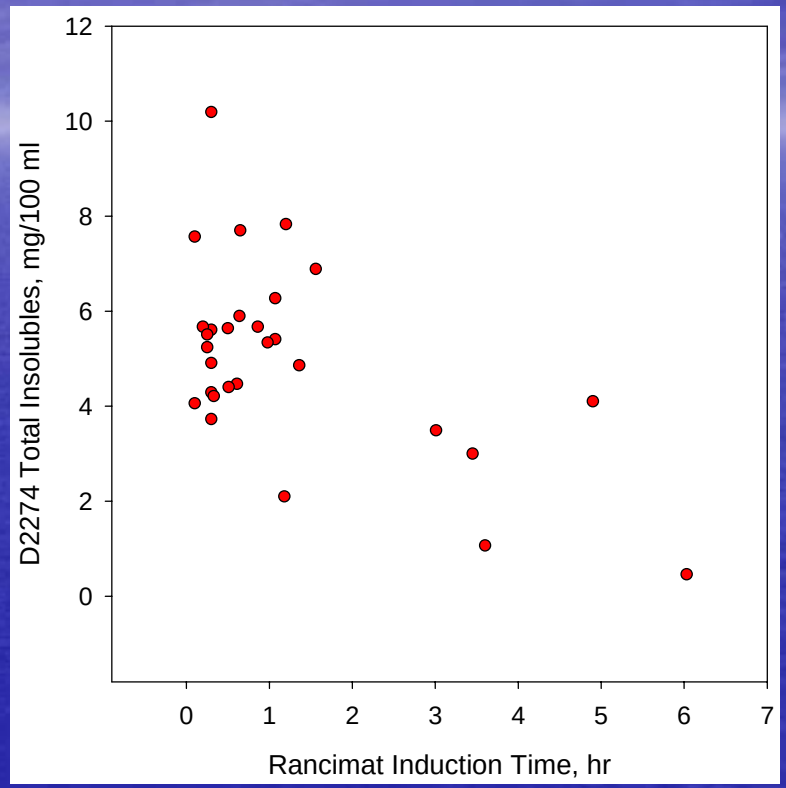
- Conclude that the average biodiesel (meeting D6751) is probably adequately stable when used as B20 with existing diesel fuel

ASTM Oxidation Stability WG

- Two methods have risen to the top
- D2274 with glass fiber filters
  - More direct measurement of deposits
  - Preferred by US engine companies and EMA
  - With possible dilution with non-polar solvent to more clearly identify effects with ULSD
- “Rancimat”, measures volatile acids after aging and produces induction period value
  - Provides measure of ‘aging reserve’
  - Preferred by European FIE and some auto makers
- One added method: Anti-oxidant concentration

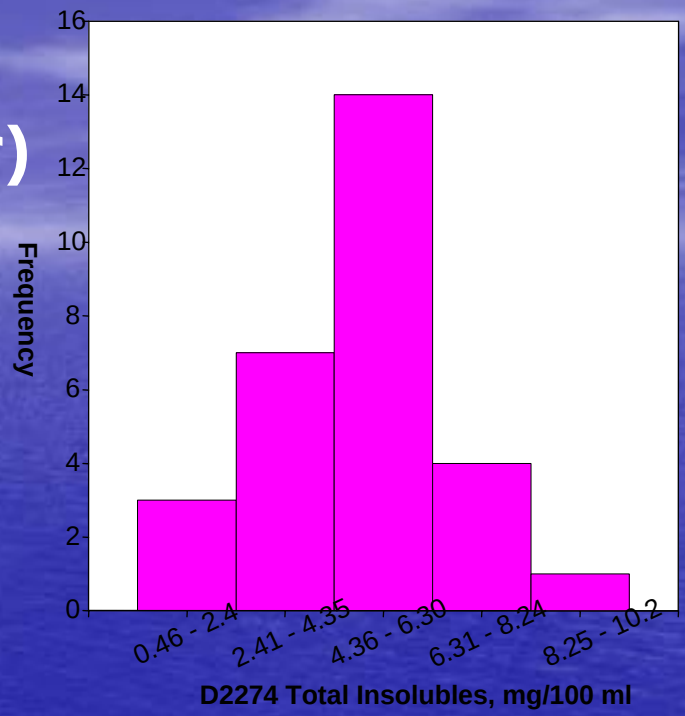
Rancimat vs Deposit Formation

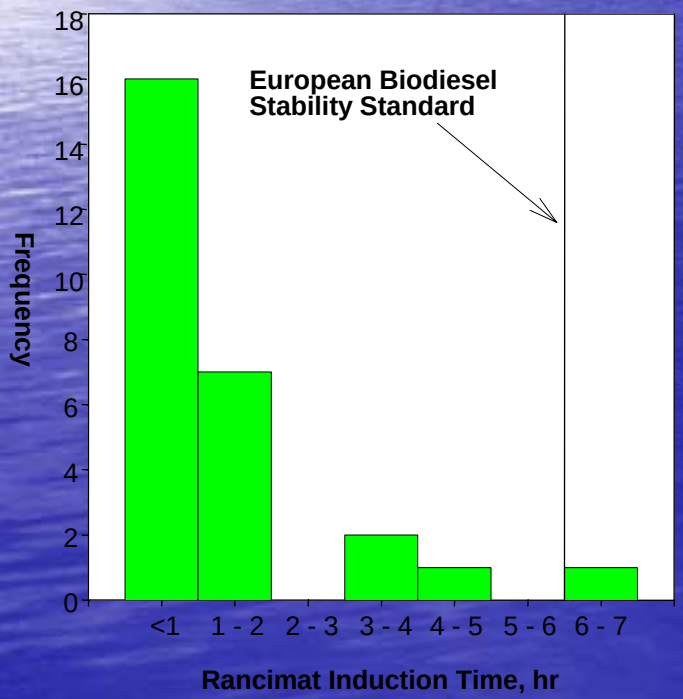
- D2274 and Rancimat results are not well correlated
  - Some samples with short induction times have low levels of deposits
  - Volatile acid formation is not representative of deposit formation
  - Certain deposit formation pathways are not oxidation dependent
- Deposit formation may be preferred - possibly modified D2274



Quality Survey Stability Results

- ASTM D2274 (95°C/Oxygen/16 hr)
- Measures deposit formation – relevant for engine operation
- Average US biodiesel produces 5 mg insolubles/100 ml





- Rancimat (EN14112)-in Europe B100 spec
- Measures induction time for volatile acid formation
- Typical US biodiesel has less than 2 hr induction time

ASTM Oxidation Stability WG

- Significant additional work is planned
  - Test method precision and comparison
    - Rancimat and D 2274
  - Ability of each method to predict benefits of anti-oxidants in reducing sediments and deposits
  - Comparison of bench scale results to actual field operations with B20 and lower blends
  - Determination if control of B100 stability is sufficient for all blend levels with all petrodiesel

ASTM Oxidation Stability WG

- ASTM Oxidative Stability Study will commence in October
- Control at the B100 level is preferred
  - Anti-oxidants are most easily added to B100
  - Blending is done at terminal
- NBB has set up BQ-9000 program for marketers
  - Monitors acid number as surrogate for stability

BQ-9000 Quality Program

- The BAC (Biodiesel Association of Canada) and the CRFA (Canadian Renewable Fuels Association) have endorsed the BQ-9000 Quality program
- Some OEM's are starting to include this requirement in their warranties

BQ-9000 Quality Program

- Demonstrate an ability to provide product that meets ASTM D 6751.
- Includes processes for corrective action and the prevention of nonconformity.
- Applies equally to producers or marketers in the Biodiesel Industry.
- Provides confidence to you and the customer that intended quality is met through demonstrated capabilities.

BQ-9000 Quality Program

- Program is designed to monitor the production of biodiesel to the ASTM D 6751 specification including:
  - Sampling
  - Testing
  - Retain Samples
  - Shipping

BQ-9000 Quality Program

- **Production Lots**
- Each lot shall be identified in a manner that corresponds to a particular volume of fuel.
- Once a lot has been identified, no additional product shall be added unless it is retested to confirm that it meets specification.

BQ-9000 Quality Program

- **Testing of Production Lots**
- First Steps
  - Take a sample for visual inspection
- Sample according to appropriate testing protocol
  - Full specification testing until confidence is achieved (7 consecutive lots)
- A portion of the production lot sample shall be retain for a minimum of 60 days.

BQ-9000 Quality Program

- A significant change in the process requires reestablishment of confidence in the system.
  - Full Specification Testing
  - Three consecutive production lots
- At least once every six months, a production lot sample must undergo full specification testing
- Generate a COA

BQ-9000 Quality Program

- Trailers and railcars should be dedicated to biodiesel service
  - If this is impractical, the producer shall provide cleanliness specification standards that address material and chemical compatibility issues and vessel cleanliness.
- Each trailer and railcar compartment shall be inspected to assure the standards have been met before the loading operation begins.

BQ-9000 Quality Program

- Trucks and railcars designed to accommodate closure and identification seals shall be used.
- At the end of the loading operation, all inlet valves to the compartments shall be closed and sealed.
  - If this is not possible, alternative procedures designed to help prevent the potential adulteration of product shall be developed and used by the producer.

BQ-9000 Quality Program

- **BQ-9000 Accredited Marketer**
- The marketer can purchase biodiesel from an NBAC accredited producer and rely on the COA generated by the producer, or
- The marketer can purchase the biodiesel from a non-accredited producer and have the testing performed to produce a valid COA

BQ-9000 Quality Program

- Product received from an NBAC accredited producer may bypass the ASTM D 6751 tests required to generate an additional COA upon receipt and be off-loaded directly into a distribution tank.
- The producer generated COA applies to this product.
- A representative sample shall be taken as the truck is off-loaded into the distribution tank.
- The sample shall be visually inspected for water, sediment, and particulate matter.