Biodiesel Fuel Quality

National Survey Results
and the
Need for Surveillance Testing

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NREL 2004 B100 Quality Survey

- 22 producers in the marketplace (25M gallons)
  - Samples obtained nationwide from biodiesel blenders (27 samples)
- ***85% of samples tested met the ASTM D6751 specification***
- Four samples failed with high levels of:
  - phosphorus (lube oil contamination?)
  - total glycerin
  - acid number

- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

NREL 2006 B100 Quality Survey

- 86 companies in the marketplace (250M gallons)
- **41% of B100 samples passed the ASTM D6751 specification**
- 30% failed total glycerin – immediate operational problems in cold weather and long term injector damage
- 20% failure rate for Na+K
  - Significant impact on emissions control systems
  - Compares to 15% failure rate in 2004 survey

- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

NREL 2007 Survey Approach

- Study conducted under guidance of Biodiesel Blend Evaluation Team (industry group of primarily engine OEMs)
- Collect B100 samples directly from producers
- Test samples for properties most likely to affect engine performance:
  - Flash point, free and total glycerin, oxidation stability, cloud point, acid value, water and sediment
- Test samples for minor compounds that could affect new or future diesel after-treatment systems:
  - Phosphorus, Group I and II metals

- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

NREL Survey Test Results

- Samples are representative of biodiesel market in US
- **89.9% of B100 samples passed the ASTM D6751 specification**
  - 94% of the volume from large producers
  - 68% of the volume from medium producers
  - 28% of the volume from small producers
- Biodiesel industry focus on quality is working

- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

Survey Sample Size

- Sample size is 70% of B100 market in 2007 or 278M gallons
  - Large producers: 89% (15 companies)
  - Medium producers: 10% (16 companies)
  - Small producers: 1% (25 companies)
- 56 out of 107 producers provided samples
- 17 out of 19 BQ-9000 producers participated
- Wide variety of feedstock compared to previous surveys
- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

Properties Off Specification

- **Free and Total Glycerin**
  - ~2M gallons off spec for FG
  - ~0.6M gallons off spec for TG
- **Acid Value**
  - 2M gallons off specification
- **Na+K and Ca+Mg**
  - <0.5M gallons off spec for Na+K
  - 2M gallons off spec for Ca+Mg
- **Oxidation Stability**
  - 8.5M gallons off specification
  - Most often from small and medium producers
- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

Survey Summary

- BQ-9000 producers have very low failure rate regardless of production volume
- Small producers failed specifications more often than medium or large producers
- Relatively high failure rate for oxidation stability, but small portion of total market (~8.5M gallons)

- Source: Teresa L Alleman, National Renewable Energy Laboratory NBB Conference

Additional Issues: Long Term Use and Product Storage

- For long term storage (not defined), the user should consider monitoring several quality parameters of the **blended fuel product** to ensure product remains in specification such as:
  – Formation of insolubles,
  – Peroxide Value (PV),
  – Total Acid Number (TAN)
  – Viscosity
  – Ester Content
  – Polymer Content
  – Microbial Contamination

- Ref: Ed English, 2008 © Fuel Quality Services, Inc.- NBB Conference 2008

Additional Issues: Long Term Use and Product Storage

- Based on the surveillance results or the presence of **excess free water present in the storage tank**, the biodiesel and biodiesel blends may need to be treated with the appropriate additive(s) to minimize on-going degradation mechanisms:
  - Anti-Oxidants
  - Metal Deactivators
  - Multifunctional Stabilizer Packages
  - Microbicides !!!!

- Ref: Ed English, 2008 © Fuel Quality Services, Inc.- NBB Conference 2008

BQ-9000

- To **promote** the commercial success and public acceptance of biodiesel

- To help **guarantee** that biodiesel fuel is produced and maintained at ASTM D 6751 levels

- Accredited Producer
- Certified Marketer

www.BQ-9000.org

ASTM D 6751-07b

Property	Test Method	Limits	Units
Calcium & Magnesium	EN 14538	5 max	ppm (ug/g)
Alcohol control			
either Flash Point	**D 93**	**130 min.**	**Degrees C**
or GC methanol	**EN 14110**	**0.2**	**% Volume**
Water and Sediment	**D2709**	**.050 max**	**% Volume**
• **Flash Point**	**D 93**	**93 min.**	**Degrees C**
Kin. Viscosity, 40C	D 445	1.9 - 6.0	$mm^2/sec.$
Sulfated Ash	D 874	0.02 max.	% mass
Sulfur S500	**D 5453**	**0.05 max (500)**	**% mass (ppm)**
S15	**D 5453**	**0.0015 max (15)**	**% mass (ppm)**
Copper Corrosion	D 130	No. 3 max.	
Cetane number	D 613	47 min.	
Cloud Point	**D 2500**	**Report**	**degrees C**
Carbon Residue	D 4530	0.05 max.	% mass
• **Acid Number**	**D 664**	**0.50 max.**	**mg KOH/g**
• **Free Glycerin**	**D 6854**	**0.020**	**% mass**
• **Total Glycerin**	**D 6854**	**0.240**	**% mass**
Phosphorous content	D 4951	0.001 max	% mass
Distillation, T90 AET	D 1160	360 max	degrees C
Na/K, combined	EN 14538	5 max	ppm (ug/g)
Oxidation Stability	**EN 14112**	**3 min**	**hours**
• **Visual App.**	**D 4176**	**Free of un-dissolved water, sediment and suspended matter**	

BOLD = BQ-9000 Critical Specification Testing ; BOLD= BQ-9000 Certified Marketer Surveillance Program

Quality Surveillance Program

- Obtain ASTM D 6751 COA from all Producers
- Take samples at B100 / B99 level
  - Biodiesel Plants, Pipeline Terminals, Bulk Distributors
- Utilize BQ 9000 Certified Marketer tests
  - Five (5) B100 Properties
  - Flash Point, Acid Number, Free Glycerin & Total Glycerin, Visual Appearance