2008 B20 Survey Results

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Acknowledgments

- Department of Energy
- National Biodiesel Board
- Engine Manufacturers Association
2004 B20 Quality Survey Results

- 22 producers in the marketplace (25 M gallons)
- Samples obtained nationwide from biodiesel blenders (27 samples)
Biodiesel Content from 2004 Survey

- 18 samples (36%) fall outside the 18-22% range
- Likely cause: poor mixing during splash blending
B20 Survey and D7467

- This B20 survey was taken as part of a cooperative effort between NREL, the National Biodiesel Board (NBB), and the Engine Manufacturers Association (EMA) to gain further information on B20 in the market (Biodiesel Blend Evaluation Team, BBET).

- ASTM D7467 (ASTM spec for B6 to B20 blends) was not released for use until October 2008.

- B20 samples were taken prior to release of D7467.

- Parameters, in addition to those proposed in D7467, were analyzed for additional information to provide data to ongoing NREL, NBB, and EMA technical efforts on biodiesel.

- D7467 was designed so B6 to B20 would always be in-spec if in-spec diesel and in-spec biodiesel were used to meet the blend.

- Recent B100 survey indicated 90% of biodiesel volume meets D6751.
Pump Labeling

- There were no pump labeling regulations in effect when the samples were taken.
- Only pumps that were labeled ‘B20’ were sampled.
- Only fleets that identified that they were purchasing and using B20 were sampled.
- As part of EISA 2007, the Federal Trade Commission (FTC) issued federal regulations requiring pump labels for biodiesel pumps, which took effect in Dec. 2008:
  - Less than 5% biodiesel content does not require a label.
  - Pumps with biodiesel content between 6% and 20% must be labeled with wording such as ‘contains between B6 and B20.’
  - Pumps over B20 must be labeled with the exact percentage of biodiesel.
- NCWM is adopting the FTC pump labeling language for biodiesel blends as part of NIST Handbook 130.
### 2008 B6-B20 Property Testing

#### D7467 Specification

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Method</th>
<th>S15 Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid number, mgKOH/g</td>
<td>D664</td>
<td>0.3 max</td>
</tr>
<tr>
<td>Viscosity at 40°C</td>
<td>D445</td>
<td>1.9-4.1</td>
</tr>
<tr>
<td>Flash point, °C</td>
<td>D93</td>
<td>52</td>
</tr>
<tr>
<td>Cloud point, °C or LTFT/CFPP, °C</td>
<td>D2500 or D4539/D6371</td>
<td>Report</td>
</tr>
<tr>
<td>Sulfur, ppm</td>
<td>D5453</td>
<td>15 max</td>
</tr>
<tr>
<td>T90, °C</td>
<td>D86</td>
<td>343 max</td>
</tr>
<tr>
<td>Carbon residue, mass%</td>
<td>D524</td>
<td>0.25 max</td>
</tr>
<tr>
<td>Cetane number</td>
<td>D613</td>
<td>40 min</td>
</tr>
<tr>
<td>Cetane index</td>
<td>D976</td>
<td>40 min</td>
</tr>
<tr>
<td>Aromatics, vol%</td>
<td>D1319</td>
<td>35 max</td>
</tr>
<tr>
<td>Ash, mass%</td>
<td>D482</td>
<td>0.01 max</td>
</tr>
<tr>
<td>Water and sediment, vol%</td>
<td>D2709</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Copper corrosion, 3 hours at 50°C</td>
<td>D130</td>
<td>No. 3 max</td>
</tr>
<tr>
<td>Biodiesel content, vol%</td>
<td>D7371</td>
<td>6-20</td>
</tr>
<tr>
<td>Oxidation stability, hrs</td>
<td>EN14112</td>
<td>6 min</td>
</tr>
<tr>
<td>Lubricity, µm</td>
<td>D6079</td>
<td>520 max</td>
</tr>
</tbody>
</table>

#### Tests Performed

<table>
<thead>
<tr>
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<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid number, mgKOH/g</td>
<td>D664</td>
</tr>
<tr>
<td>Flash point, °C</td>
<td>D93</td>
</tr>
<tr>
<td>Cloud point</td>
<td>D2500</td>
</tr>
<tr>
<td>Sulfur, ppm</td>
<td>D5453</td>
</tr>
<tr>
<td>Group I and II metals, ppm</td>
<td>D7111</td>
</tr>
<tr>
<td>Karl Fisher, ppm</td>
<td>D6304</td>
</tr>
<tr>
<td>Cetane number</td>
<td>D6890</td>
</tr>
<tr>
<td>Particulate contamination, ppm</td>
<td>D6217 or D7321</td>
</tr>
<tr>
<td>Interfacial tension, mN/m</td>
<td>D971</td>
</tr>
<tr>
<td>Ash, mass%</td>
<td>D482</td>
</tr>
<tr>
<td>Free glycerin, mass%</td>
<td>In-house method</td>
</tr>
<tr>
<td>Total glycerin, mass%</td>
<td>In-house method</td>
</tr>
<tr>
<td>Biodiesel content, vol%</td>
<td>D7371</td>
</tr>
<tr>
<td>Oxidation stability, hrs</td>
<td>EN14112</td>
</tr>
</tbody>
</table>
Sample Collection

- 32 samples collected across the United States in late spring and early summer
- Sample locations chosen to cover widest geographic range possible
- Collected from fleets using B20 or from public pumps
  - Only selected pumps stating product was B20
Results
(Note: D7467 was not in effect when samples were collected.)
Blend Percentage Results

- 60% of samples were nominally B20
- Sample distribution much different than for 2004 results
  - All fleet samples were nominally B20
- Much fewer samples above B20
- Most off-spec samples were less than B20
  - Typically B11, B5, and B2
Induction Period Stability

- D7467 not required when samples were collected
- 74% within D7467 for B20 Induction Period

Average B20 Induction Period = 8.4 hrs

Testing was stopped after 12 hours

Specification is 6 hrs, min

Sample ID

Fleet Samples
Acid Value Results

- 97% within D7467 for B20

**Acid Value, D664, mgKOH/g**

- Samples at 0.05 mgKOH/g are below detection limit
- Specification Limit = 0.3, max

**Sample ID**

- Nominally B20
- Less than B20
- Greater than B20

**Fleet Samples**

- 97% within D7467 for B20
ASTM SC06 Modifications to D664

- Modifications are being balloted at ASTM
- Allows better discrimination at lower acid values

The samples were analyzed using the current method upon collection. There was a delay of several weeks before they were analyzed using the modified method.

Note samples that are below detection limit on D664 have been assigned a value at that limit of 0.05.
Cloud Point

Average B20 CP = -12.6°C

- Nominally B20
- Less than B20
- Greater than B20

Fleet Samples
Flash Point

- Nominally B20
- Less than B20
- Greater than B20

- 52°C minimum flash point if CP is >-12°C
- 38°C flash point if CP is <-12°C
# Other D7467 Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM Method</th>
<th>Range, Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfur, ppm</td>
<td>D5453</td>
<td>3.7-16.2, 7.3</td>
</tr>
<tr>
<td>Derived cetane number</td>
<td>D6890</td>
<td>45-56, 49</td>
</tr>
<tr>
<td>Ash, mass%</td>
<td>D482</td>
<td>BDL, BDL</td>
</tr>
</tbody>
</table>

* BDL: below detection limit
Results – Non-D7467 Properties
Karl Fisher Moisture

- Current biodiesel and diesel specifications measure ‘free water’ using a centrifuge method for ‘water and sediment.’
  - D6751, D7467, D975
  - Free water is the property of most concern as this can increase water-related issues.
- EMA members desired information on both the free water and the entrained/soluble water.
- Karl Fisher moisture measures both the free water AND any entrained/soluble water (not part of the current specifications for biodiesel, biodiesel blends, or petrodiesel).
- B100 can hold slightly more water in solution than most petrodiesel.
- There is an ongoing working group within the ASTM Biodiesel Task Force looking into replacing the current water and sediment test method with a separate measure for KF moisture and a separate measure for fuel particulates.
Karl Fisher Moisture

B20 average KF water = 130 ppm

- Nominally B20
- Less than B20
- More than B20

Fleet Samples
Interfacial Tension

Average B20 IFT = 12.3 mN/m

- Nominally B20
- Less than B20
- Greater than B20

Fleet Samples
Group I and II Metals

- Group I and II metals are controlled at the B100 level:
  - Sulfated ash of 0.02%
  - 5 ppm maximum of combined Na/K
  - 5 ppm maximum of combined Ca/Mg

- Method not approved for biodiesel used to measure these metals in the low concentrations expected in the blends

<table>
<thead>
<tr>
<th>Property, ASTM D7111</th>
<th>Range, Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg, ppb</td>
<td>&lt;100 (detection limit for all samples)</td>
</tr>
<tr>
<td>K, ppb</td>
<td></td>
</tr>
<tr>
<td>Ca, ppb</td>
<td>&lt;100 to 652, 219</td>
</tr>
<tr>
<td>Na, ppb</td>
<td>&lt;500 to 2000*, 606</td>
</tr>
</tbody>
</table>

* 1 sample would be off-spec at B100 for Na.
Conclusions – D7467 Properties

- B20 samples were collected from around the United States, from public pumps and fleets as part of joint effort of NREL, NBB, and Engine Manufacturers Association.
- D7467 was not enacted when these samples were taken.
- 60% of blends were nominally B20.
  - Distribution of non-B20 samples was different from that in last survey, with much less fuel above 20%; indicates less splash blending type issues but still room for improvement.
  - High percentage below B20 indicates need for improvement in pump label management and enforcement.
- Average cloud point (CP) = -12.6°C (9°F).
  - No CP specification; determined by geography, time of year, customer.
- Average flash point = 59°C (138°F); none outside D7467.
- 74% of samples met D7467 induction period stability requirements for B20.
- Only 1 sample off-spec for D7467 acid value.
  - Indicates fuel had not degraded to the point of being problematic even though the induction period was below current D 7467 specification level.
Conclusions – Non-D7467 Properties

- Interfacial tension average was 12.3 mN/m.
- Average Karl Fischer moisture was 130 ppm.
  - ULSD can average 50-150 ppm.
- Elevated Na content was observed in 1 sample, indicating the B100 was not on-spec (blend was off-spec for other parameters as well).
- Ca, Mg, and K were extremely low and near detection limits.
Recommendations

• ASTM has issued the new D7467 specifications for B6 to B20 blends, and FTC has issued nationwide pump labeling requirements for biodiesel blends.

• Fuel enforcement agencies now have specifications to help enforce quality of biodiesel blends.

• Improvement is needed to ensure that the amount of biodiesel being advertised is what is really being provided to the customer (although this seems to have improved since the last survey).