

# B I O D I E S E L

## M A G A Z I N E

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### QUALITY

## Field Testing Equipment Enhances Quality Assurance

By Robert Young



According to the National Biodiesel Board, biodiesel production in the United States reached 450 million gallons in 2007, up significantly from 2005 and 2006. As a result of minimum bio-fuel blend requirements set forth in the Energy Independence and Security Act of 2007, biodiesel usage is expected to expand to 500 million gallons in 2009 and 1 billion gallons by 2012.

State and city mandates are also driving the demand for biodiesel. Minnesota recently passed a bill requiring that the percentage of biodiesel in diesel fuel increase from 2 percent to 20 percent by 2015. Pennsylvania and Massachusetts have also passed significant legislation promoting biodiesel use. One year ago, Portland, Ore., mandated that all diesel sold within the city contain 5 percent biodiesel. San Francisco's entire city-owned diesel vehicle

The field test kits for B100 are a quick check in the field for detecting traces of catalyst, mono-, di- or triglycerides, soaps, acids and oxidized fuel.

fleet, which includes city buses, ambulances and street sweepers, uses B20.

Interest in biofuels is also building in school districts, many of which are running their buses on B20 in an effort to reduce pollution and save money. One recent story documents how a Future Farmers of America chapter in West Virginia took advantage of state government grants to begin producing its own biodiesel to power the school district's bus fleet.

Though biodiesel is becoming more

popular, a recent study from the Woods Hole Oceanographic Institute gives reason for caution. The study found that biodiesel blend percentages can vary significantly without producer or consumer knowledge. A *Boston Globe* article discussing the study's results noted that "fuels advertised as having 20 percent biodiesel actually had anywhere from 10 (percent) to 74 percent." In other words, due to lack of testing and verification, consumers may unintentionally be misled at the pump.

These increases in government activity,

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PHOTO: ELIZABETH SLAVENS, BBI INTERNATIONAL

Some field test kits, such as the pHLip test, provide results in as little as 10 minutes.

along with documented inaccuracy in biodiesel blending, create an urgent need for reliable and cost-effective testing of biodiesel fuel components. “It’s critical that the fuel (biodiesel) meet the specifications,” said National Biodiesel Board spokeswoman Jenna Higgins. “An off-specification batch can create problems and a bad batch of fuel can be devastating (for the growth of our industry).” As the market for biodiesel grows, thorough testing will be essential to ensure the safe and optimal performance of our cars, trucks and buses, as well as the continued success of the biodiesel industry.

### Assessing Fuel Quality

Fuel quality is the key issue for any biodiesel producer or distributor. Fuel is transported through numerous channels before it gets to the consumer, creating many points along the supply chain at which fuel quality may be compromised. Fuel quality can be compromised and deteriorate as it is introduced, stored, blended, or further distributed and sold. As with petroleum diesel, problems in fuel quality can result in clogged filters and injectors, rough engine operation and wear, ruined fuel injection pumps, crank case oil deterioration and long-term engine damage.

Identifying the presence of several con-

taminants is critical to ensure the final blend meets proper specifications. These contaminants include water, methanol, total and free glycerin, microbial contamination and acid number.

To check quality and blend percent, field testing equipment has been developed and is in use. Products range from the qualitative pHLip tests and Fleet Biodiesel testing methods, to the more quantitative Wilks Infracpec and the i-SPEC Q-100 handheld analyzer using impedance spectroscopy technology. Below is a brief overview of the operations and capabilities of these products.

**pHLip Tests:** The field test kits for B100

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are a quick check in the field for detecting traces of catalyst, mono-, di- or triglycerides, soaps, acids and oxidized fuel. While no technical expertise is required to operate the kit, interpreting observations can be difficult. The basic test takes 10 minutes and involves adding B100 to a solution in a vial, flipping and redistributing the solution into two phases, followed by visual observation. If soap is present it is seen as turbidity. Glycerides and oxidized fuel are seen as concentration at the interface. Fatty acids or triglycerides are seen as turbidity. The color becomes progressively purple with increased acidity. As described by their manufacturers, these tests work as a “firewall” and are to be followed up with lab tests.

#### The 3/27 (Jan Warnqvist) Test:

This basic field test, which can be performed on washed or unwashed fuel, requires no technical expertise and is based on the differences in solubility of biodiesel and oil in methanol. The name is derived from the 1:9 ratio of biodiesel to methanol used for this test. A passing sample, which indicates acceptable conversion, will remain clear with only slight color change and no separation of layers. A failing sample often appears as cloudiness or as a pool of unreacted feedstock. It is suggested to monitor the sample for 30 minutes before determining a passing sample. Results are subjectively examined. Because this test is based on solubility, it is sensitive to temperature. The recommended test temperature is 68 plus or minus 2 degrees Fahrenheit.

**Fleet Biodiesel Test Kits:** Fleet Biodiesel Inc. provides test kits that require no technical expertise to operate. Again, the biodiesel or blend is added to vials containing different solutions. These kits are de-

#### Specification for B100--ASTM D 6751

Property	ASTM method	Limits	Units
Calcium and magnesium, combined	EN 14538	5 maximum	parts per million (ug/g)
Flash point (closed cup)	D 93	93 minimum	degrees Celsius
Alcohol control (one of the following must be met)			
1. Methanol	EN 14110	0.2 maximum	percent volume
2. Flash point	D 93	130 minimum	degrees Celsius
Water and sediment	D 2709	0.05 maximum	percent volume
Kinematic viscosity (40 degrees Celsius)	D 445	1.9-6.0	millimeters per second squared
Sulfated ash	D 874	0.02 maximum	percent mass
Sulfur			
1. S 15 grade	D 5453	0.0015 maximum	percent mass
2. S 500 grade	D 5453	0.05 maximum	percent mass
Copper strip corrosion	D 130	No. 3 maximum	
Cetane	D 613	47 minimum	
Cloud point	D 2500	Report	degrees Celsius
Carbon residue 100 percent sample	D 4530	0.05 maximum	percent mass
Acid number	D 664	0.5 maximum	milligrams of KOH per gram
Free glycerin	D 6584	0.02 maximum	percent mass
Total glycerin	D 6584	0.24 maximum	percent mass
Phosphorous content	D 4951	.001 maximum	percent mass
Distillation, T90 AET	D 1160	360 maximum	degrees Celsius
Sodium/Potassium, combined	EN 14538	5 maximum	parts per million (ug/g)
Oxidation stability	EN 14112	3 minimum	hours
Workmanship	free of undisclosed water, sediment and suspended matter		

SOURCE: NATIONAL BIODIESEL BOARD

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signed for use with all blends of fuel from B2 through B100. Test kits similar to the pHip test are available for B5/B20 and a separate B100 kit is available for water, visual clarity and acid number. A test kit for yeast/mold and anaerobic bacteria is also available. The blend percent tests are threshold tests indicating if a B5 blend contains more than 10 percent biodiesel and B20 contains more than 30 percent. The total glycerin field test kit has visual ranges for 0.05 percent to 0.5 percent.

**Wilks Infracpec:** This small infrared instrument can make pass/fail determinations of total glycerin with an error of 0.03 percent in the range of 0.24 percent total glycerin. All readings above 0.26 percent fail and readings below 0.22 percent pass. There are factors which limit the accuracy and precision of this method, but it can be used for pass/fail monitoring. This method is rapid, yielding results in less than one minute. This instrument has also been used to determine blend percent and was compared to the Fourier transform infrared spectroscopy EN method 14078, demonstrating results accurate within 5 blend percent below 50 percent and within 15 blend percent above 50 percent.

**The Paradigm i-SPEC Q-100:** The trademarked i-SPEC handheld analyzer can be used by anyone from the plant through the distribution chain. A sample is added to a cartridge and the analyzer produces a set of results. This handheld analyzer tests the quality, as well as the blend percent, using impedance spectroscopy.

Impedance spectroscopy has demonstrated excellent correla-

tion to traditional reference methods. The i-SPEC can simultaneously measure the blend percent and original total glycerin in B100 used for blends of 5 percent to 50 percent within minutes, and also tests for total glycerin, methanol and acid number in B100 samples. This is a mobile testing unit designed for ease of use and rapid test results.

These results are quantitative for blend percent within 1 percent to 2 percent and for total glycerin to 0.07 percent. The determination for methanol is pass/fail for within or out of ASTM spec, as is the case for acid number. No interpretations are necessary, nor are subjective observations used to evaluate results.

## Conclusions

No single method will satisfy all the testing needs and test for all the different critical parameters to ensure that biodiesel meets the required standards. Quick qualitative methods with pass/fail capabilities have been important tools in testing frequently and throughout the distribution chain. New methods with increased quantitative capabilities and accuracy provide advantages while offering the ease of use, portability and ruggedness for plant and field testing. ■

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