Evaluation of the i-SPEC® Q 100 Handheld Biodiesel Analyser in Ten Laboratories

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A new handheld biodiesel analyser was introduced in 2009 to screen for key critical parameters in biodiesel quality, both for B100, as well as for blends. Fuel quality is the key issue faced by any biodiesel producer, distributor, fleet manager, or fueling station. 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. Biodiesel quality can deteriorate as it is introduced, stored, diluted, or further distributed and sold. 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.

The Paradigm i-SPEC® Q 100:

The new Paradigm Sensors' i-SPEC® Q 100 handheld biodiesel analyser can be used by anyone from the plant throughout the distribution chain. This analyser uses Impedance Spectroscopy (IS), which has demonstrated excellent correlation to traditional reference methods (Dr. R. Hirthe, AOCS presentation 2008) to test the quality and percent of biodiesel blends. The i-SPEC® is the translation of the laboratory method to a portable handheld screening tool, which can simultaneously measure the blend percent from 2-99% and original total glycerin in biodiesel used for blends of 6-99% and also tests for total glycerin (TG), methanol, and acid number (AN) in B100 samples. This portable testing unit is extremely easy to use and provides results within minutes.

Impedance Spectroscopy

The use of AC impedance measurements to characterise the electrical properties of materials systems provides an alternate means of measuring composition, as well as critical physio-chemical attributes. This methodology has been successfully applied to a variety of fluids, including multiple parameter determinations for biodiesel.

In initial laboratory studies, IS produced results which are quantitative for blend percent within 1-2% and for total glycerin to 0.04%. The determination for methanol and acid number is reported as "pass" or "fail" (within or out of spec) with an advisory quantitative value provided . No interpretations are necessary, nor are subjective observations used to evaluate results.

Figure 1: Paradigm Sensors' i-SPEC® Q100 Handheld Biodiesel Analyser

Correlation of IS Data to ASTM Physio-Chemical Parameter Values

The following Reference Methods were used to evaluate IS capabilities:

Blend Concentration
- Mid Infrared Spectroscopy (ASTM D7371)

Total Glycerin (0.03 - 0.7 %)
- Gas Chromatography (ASTM D6584)
- SafTest

Acid Number (0.2 – 3.5)
- Titration (ASTM D664)
- SafTest

Methanol
- Headspace-Gas Chromatography (EN 11410)
- IR Correlations to EN 11410

Beta Evaluation Program: Report 1

We now discuss the results of a beta evaluation program in ten laboratories with the portable embodiment of this technology. Repeatability, accuracy and use characteristics were evaluated. This report focuses on the parameters blend percent, TG of B100 and TG of blend stock for Bxx blends.

Four B100 samples and five blends were evaluated in 8 out of 10 laboratories. Additional B100 samples and blends were provided by participating laboratories. Forty-five B100 samples were studied, along with 35 blends with petroleum.

The feedstocks represented by these materials included soy, corn, canola, chicken, yellow grease, brown grease, algal as well as others. B100 materials included samples within and out of specification for one, two or three parameters. Problematic samples were fully analysed in the laboratory.

Initial Studies using Laboratory IS demonstrated excellent correlation to ASTM and EN reference methods.

Blend Concentration Correlation of the Reference Standard to IS In the Laboratory

Figure 2 demonstrates that blend concentration determined from IS measurements is very well correlated (r^2= 0.998) with that found using the ASTM FTIR standard. Upper and lower 95% prediction limits for IS blend concentration are also shown. The error in the IS derived values is within the stated lab-to-lab reproducibility of the ASTM standard, which is on the order of +/- 1.6 volume % at the B20 level.

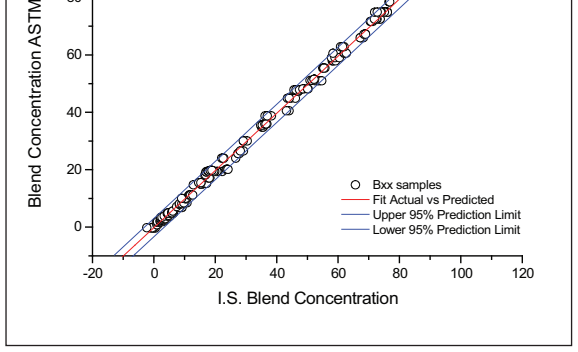

Figure 2: Blend Concentration ASTM D7371 versus IS Blend Concentration

When this study was conducted with the i-SPEC, the correlation was 0.98 to the IR method for 35 samples with blend percent from 0-90%, as shown in Figure 3.

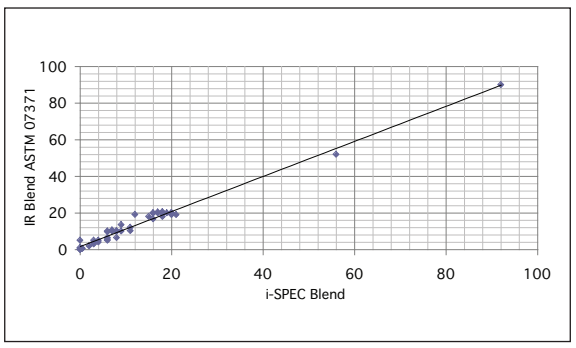

Figure 3: Blend Concentration ASTM D7371 versus i-SPEC Blend Concentration

The repeatability of the determination of blend percent for 5 samples in 8 laboratories was also determined. As is shown in Figure 4, the mean blend percent determined by i-SPEC is plotted versus the IR determination of blend percent with the standard deviation included as a bar on the graph.

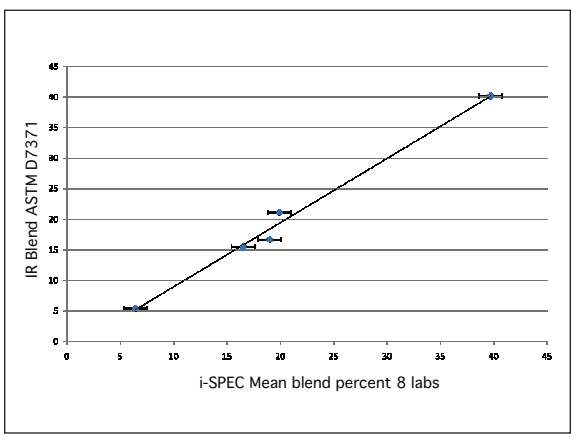

Figure 4: Repeatability of i-SPEC Blend Percent in 8 Laboratories

Correlation of the Total Glycerin (TG) Concentration Reference Standard Method to IS Spectroscopic Data on TG in the Laboratory

For B100, this Total Glycerin is one of the most critical specifications to meet, indicating the biodiesel was fully processed. Figure 5 illustrates that total glycerin content determined from IS measurements is very well correlated (r^2= .93) with that found using the ASTM D6584 GC standard method. The upper and lower prediction limits of 95% confidence for IS predicted TG concentrations are also shown. These errors in the IS derived values are within the stated reproducibility of the ASTM standard (the latter is on the order of 50% at the specification limit of 0.24 mass % and increases to 75% at 0.05 mass %).

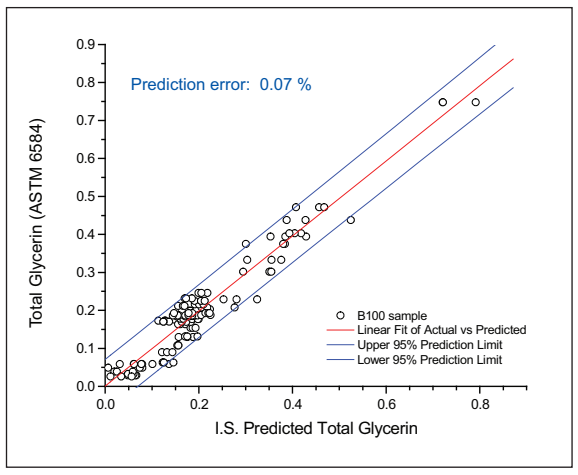

Figure 5: Total Glycerin ASTM D6584 versus IS Predicted Total Glycerin

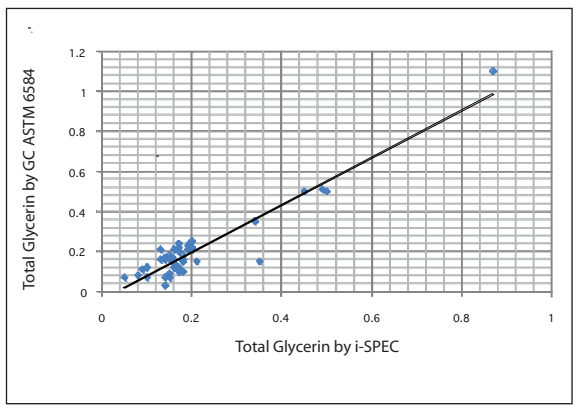

Figure 6: Total Glycerin ASTM D6584 versus i-SPEC Predicted Total Glycerin

When this TG study was conducted with the i-SPEC, the results were well correlated to the GC method, per Figure 6. The repeatability of the TG determination for 4 samples in 8 laboratories was also determined. As is shown in Figure 7, the mean TG value determined by i-SPEC is plotted versus the GC determination of TG with the standard deviation included as a bar on the graph.

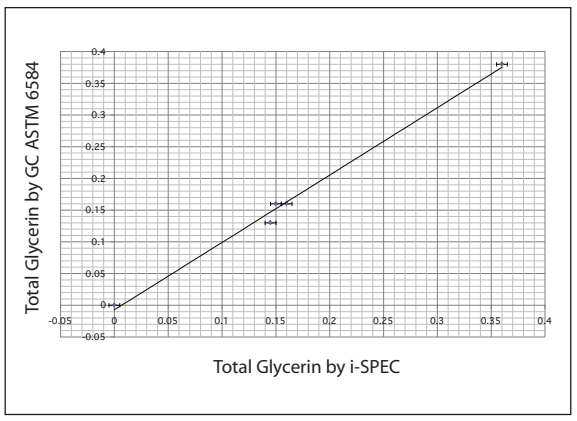

Figure 7: Repeatability of TG determination of Four samples in Eight Laboratories

Figure 8 reveals that total glycerin can also be determined in Bxx blends via IS when the blend TG values are calculated from ASTM D6584 - GC standard measurement of the respective B100 blend stocks. This allows an equivalent B100 blend stock value to be estimated for Bxx samples when blend concentration is also determined (B6 and above).

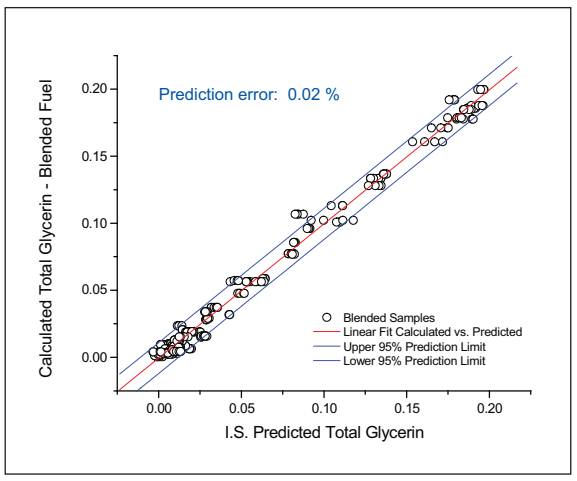

Figure 8: Calculated Total Glycerin in Blends Using IS Predicted Total Glycerin

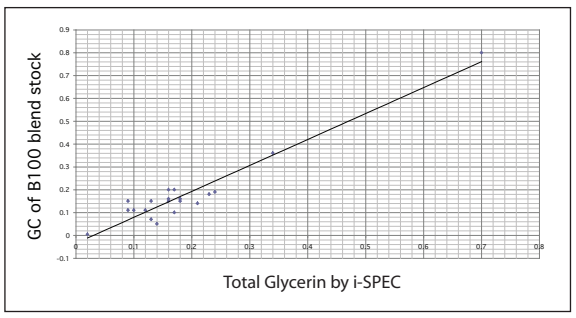

Figure 9: Calculated Total Glycerin in Blends Using IS Predicted Total Glycerin

In Figure 9 the results of the Total Glycerin in Blends using the fractional TG determination and the Blend Percent is presented. The sensitivity of TG detection is reflected in the good correlation of the value to the TG of the starting B100 determined by GC.

Interferences

We did find certain samples and properties that contributed to interferences in these measurements. Further study of these materials may allow IS identification of these properties:

1 High mineral content: Handheld properly fails sample, but reported numbers are imprecise.
Requires further study

2 Out-of-range TG, AN and methanol: Handheld properly fails sample, but reported numbers are imprecise

3 High viscosity may interfere with proper cartridge fill

4 Exotic feedstocks not well characterised, but may have less effect on handheld than other methods

- IS measurement of ethyl esters requires further study
- Distilled biodiesel may report under B100. Requires further study.

Conclusions

The i-SPEC® Q 100 is fully able to determine blend percent in blends from 2-99% and total glycerin in B100 from 0.02-0.5%. The repeatability, accuracy and ease of use of the i-SPEC make it an essential analyser for those interested in assuring the quality of biodiesel produced and distributed in today's market.