

# MEASURING BIODIESEL

ASTM 7371 and EN 14078 with the Agilent Cary 630 FTIR



Figure 1a. The the 5 bounce ZnSe ATR



Figure 1b. The TumbIIR

## Introduction

Measuring the amount of biodiesel in diesel fuel is well characterized by FTIR. There are two established methods for taking these measurements, ASTM 7371 and EN 14078, each with their own set of parameters. Both of these methods can be easily run on the Cary 630 FTIR by simply changing the sampling interfaces and loading the appropriate method. An overview of both methods is discussed below.

### ASTM 7371

This method allows the amount of biodiesel in diesel to be measured from 0-100%. To do this, three different calibration curves, one for each different range of biodiesel, 0-10%, 10-30%, and 30-100% are required. Each calibration curve also needs standards from three different cetane ranges (ultra-high, high, and low). The results are then processed using a partial least squares model. All of these measurements must utilize a multi-bounce ATR.

### EN 14078

This method allows for the amount of biodiesel in diesel to be measured from 0-10%. The method makes use of a 200  $\mu\text{m}$  pathlength cell to measure the carbonyl band at 1745  $\text{cm}^{-1}$ . The peak height is extracted and a linear fit is applied.

### Different methods, different interfaces

The ASTM method specifies measurement by an ATR sample interface while the EN method specifies measurement by transmission. The Cary 630 has an interface for each of these methods, the 5 bounce ZnSe ATR (Figure 1a) satisfies the ASTM method and the TumbIIR (Figure 1b) set to 200  $\mu\text{m}$  complies with the EN method.

Figure 2. Setting Component Parameters



The proprietary TumbIIR is a unique accessory to Agilent. It replaces the use of cumbersome IR transmission cells, eliminating errors that can be caused by air bubbles and dramatically decreasing the time and cost per analysis. As shown in Figure 3, using the TumbIIR is a simple three-step process that provides answers in seconds and increases productivity and data accuracy. A 200  $\mu\text{m}$  TumbIIR is equivalent in sampling geometry and pathlength to a 200  $\mu\text{m}$  transmission IR cell, thus complying with the EN 14078 method.

### Three steps to analysis with the TumbIIR



**Figure 3.** Using the TumbIIR is a simple three-step process

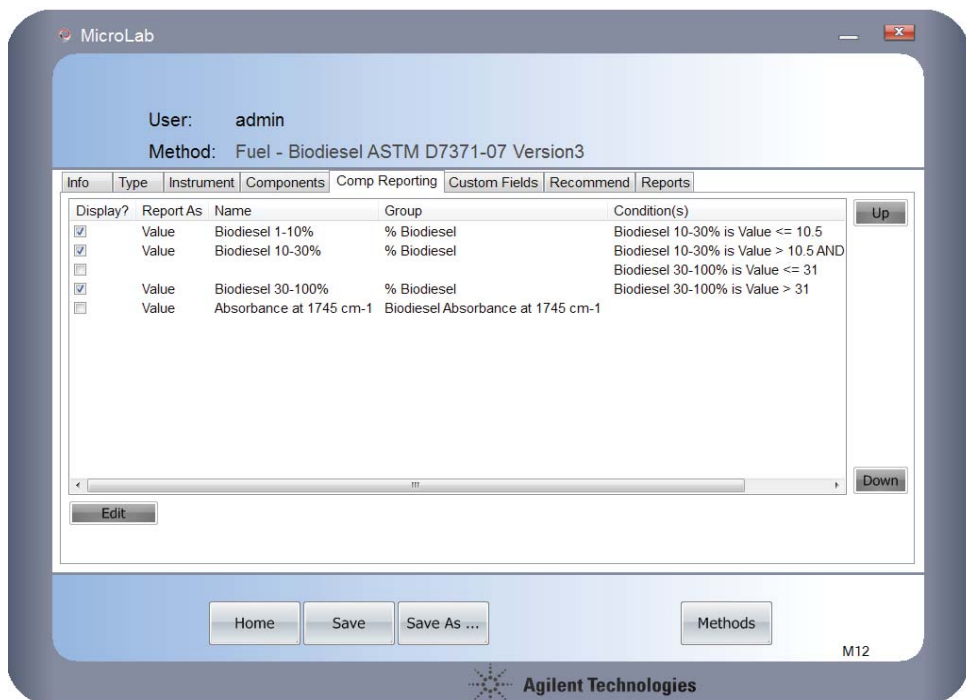
Both the TumbIIR and the 5 bounce ZnSe ATR accessories slide on to the front of the Cary 630 engine, and lock in place with no alignment required. Software recognition ensures that the proper accessory is in place for each method. In fact, the software will not let you run samples unless the accessory and method match, keeping users from making simple mistakes.

### Complex calibrations made simple

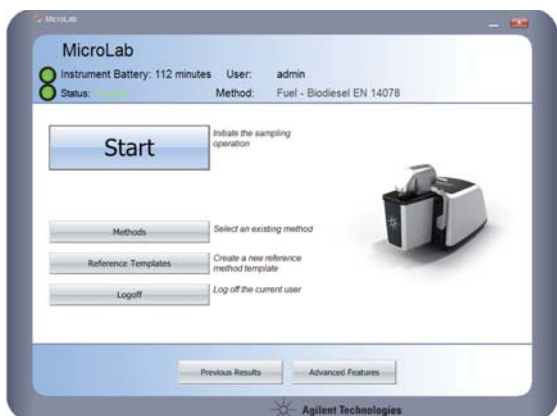
The ASTM method calls for three different calibration curves to be used in order to properly calibrate the system over the entire range. The conditional reporting built into MicroLab PC makes this seamless and transparent to the operator.

MicroLab PC automatically selects the correct calibration curve and then displays the results to the operator (Figure 4). The PLS models are loaded directly into MicroLab PC and the calculations are then performed automatically within the software.

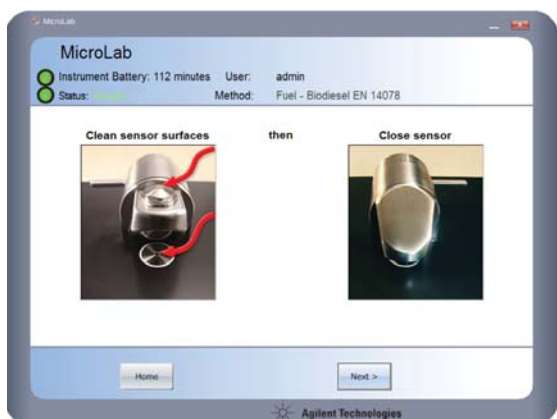
The software walks the user through the methods using an intuitive, pictorial interface. Whichever method is chosen, the software will guide users through the analysis, including built in tests and feedback to eliminate user errors.



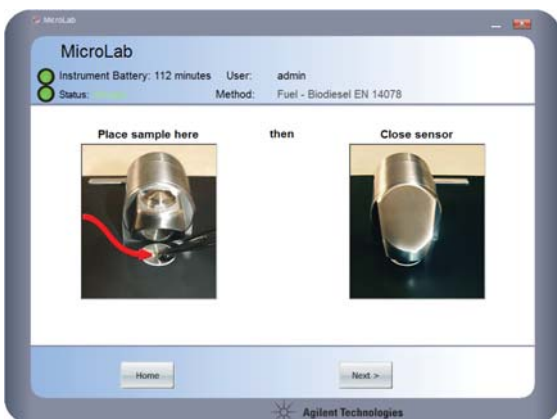
**Figure 4.** Built-in logic routines to select appropriate calibration curve



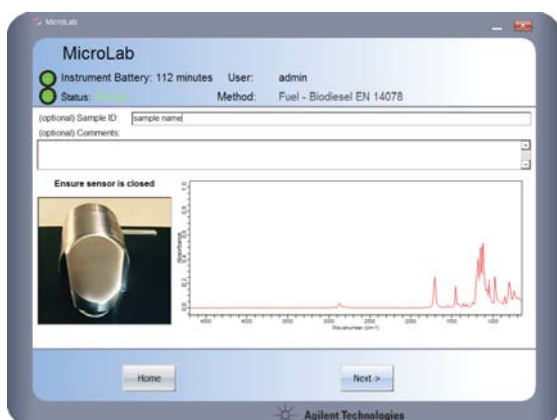
**Figure 5.** From the home screen, simply press the flashing Start button to begin



**Figure 6.** The pictorial software shows users where to clean the sensor surfaces



**Figure 7.** Users are shown where to place the sample



**Figure 8.** View live spectrum and enter sample name

#### In 4 mouse clicks, you can go from sample to answer

The design of the MicroLab PC software is to maintain the same workflow regardless of the method or the sample interface.

The method is tied to the sample interface, so for EN 14078, a TumbIIR module is added onto the Cary 630 engine. The operator simply clicks the flashing start button (Figure 5).

The software then prompts the user to clean the sensor surfaces to make sure there is no carryover (Figure 6).

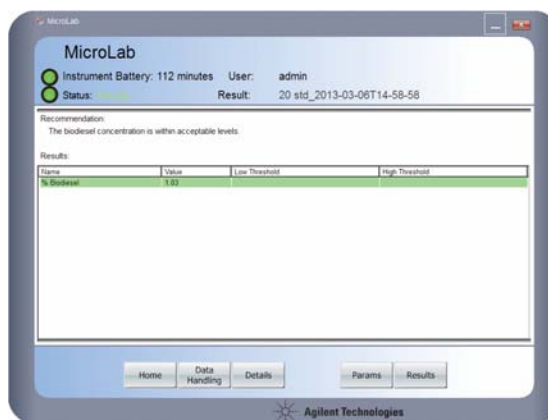
The system will then automatically check to ensure that the user cleaned the surface sufficiently and if they did not, it will prompt them to re-clean the surfaces until they pass this cleanliness check. Once the surfaces are cleaned, a background is automatically performed.

The system then visually shows the operator where to add the sample (Figure 7).

The user can then enter a sample name and see the live spectrum (Figure 8). If no sample is present, or loaded incorrectly, the software will tell the operator that there is an error loading the sample and ask them to load the sample again.

Once the user clicks on next, the calculations are performed automatically in the background and the answer is displayed onscreen.

The color-coded results are very simple to read (Figure 9). In this case, the result shown is green as this method specifies that the value must fall between 1-8%, and in this instance is 1.03. MicroLab PC allows users to easily adjust this range and associated color. For example, you can set a three color system, Green, Yellow and Red, for your method. This way the operator not only gets the numerical value, but a simple visual cue about the results. The recommendation section above the answer can be programmed to tell the operator what to do with the sample based on the results.






**Figure 9.** Color coded results are automatically displayed

## Agilent turn-key Biodiesel Analyzers

The Cary 630 is the most suitable choice for users needing to measure biodiesel content by both the ASTM and EN methods, or for users doing other types of analysis in addition to biodiesel measurement. Agilent also offers two analyzers for dedicated measurement of biodiesel by FTIR according to the ASTM or EN methods that are suitable for both routine and mobile measurement.

The Agilent portable 4500 FTIR and 5500 FTIR provide single, turn-key solutions for measuring biodiesel. These analyzers come fixed with either a multi-bounce ATR or TumbIIR sampling interface. The 4500 FTIR can be battery operated, enabling it to be taken into the field for at-site analysis. The 5500 FTIR is a robust, industrial FTIR analyzer that can be used either in the lab or on the manufacturing floor. With both the 4500 and 5500, you receive validated EN and ASTM methods, eliminating the need to develop your own calibration curves which can be a significant process. (NOTE: The 4500 and 5500 calibrated method files are not validated for nor transferable to the Cary 630.) A summary of Agilent's FTIR biodiesel solutions is below.

Instrument	Description	
Agilent Cary 630 FTIR	ZnSe Cary 630 FTIR engine with interchangeable ZnSe multibounce ATR and TumbIIR accessories. No calibrated method files.	 <p data-bbox="927 814 1198 846">Cary 630 with the TumbIIR</p> <p data-bbox="1232 814 1438 867">Cary 630 with the 5 bounce ZnSe ATR</p>
Agilent 4500 portable FTIR Biodiesel Analyzer	ZnSe FTIR system with choice of either a fixed multibounce ATR or TumbIIR. Comes with calibrated method files.	 <p data-bbox="1019 1119 1333 1150">Agilent 4500 with the TumbIIR</p>
Agilent 5500 FTIR Biodiesel Analyzer	ZnSe FTIR system with choice of either a fixed multibounce ATR or TumbIIR. Comes with calibrated method files.	 <p data-bbox="1062 1413 1338 1444">Agilent 5500 with the ATR</p>

## Conclusion

Agilent provides a full range of FTIR products for measurement of biodiesel in diesel fuel by both the ASTM D7371 and EN 14078 methods. The Cary 630 provides users with versatility for measurement of both methods by simply changing the sample interface and loading the appropriate method. The 5500 FTIR provides a complete solution including calibrated methods for either standard, and the 4500 FTIR provides the same for field applications. All products provide a unique combination of proven performance, easy-to-use software and an innovative sample interface to ensure optimum results.

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