

## NREL'S CAPABILITIES IN **BIO-OIL** CHARACTERIZATION

**NREL has pioneered reliable analyses for biomass-derived liquids produced from thermochemical conversion, such as pyrolysis bio-oils.**

Based on NREL's long history of working with bio-oils, our researchers recently developed the first standardized analytical methods that provide chemical information on bio-oil samples. Industry partners and researchers use this reliable information to inform process development and enable deployment. We also perform custom analysis tailored to your needs. Bio-oil research highlights at NREL include:

- Four validated bio-oil characterization laboratory analytical procedures
- Procedures available on [www.nrel.gov/bioenergy](http://www.nrel.gov/bioenergy) in "Data & Tools"
- Carbonyl titration method published as ASTM E3146
- New bio-oil accelerated aging test based on carbonyl titration.



Top Photo: A fast pyrolysis bio-oil sample at NREL. Photo by Dennis Schroeder, NREL 20404

Bottom Photo: An NREL researcher performs an NMR analysis. Photo by Dennis Schroeder, NREL 19988



## CORE CAPABILITIES AND APPLICATIONS

### CARBONYL TITRATION

NREL developed and validated a potentiometric titration method quantifying total carbonyl content. Carbonyls are known to cause coke formation during upgrading and lead to bio-oil aging at room temperature. This method offers a simple and reliable metric for both researchers and industry.

### PHOSPHOROUS-31 NUCLEAR MAGNETIC RESONANCE ( $^{31}\text{P}$ NMR)

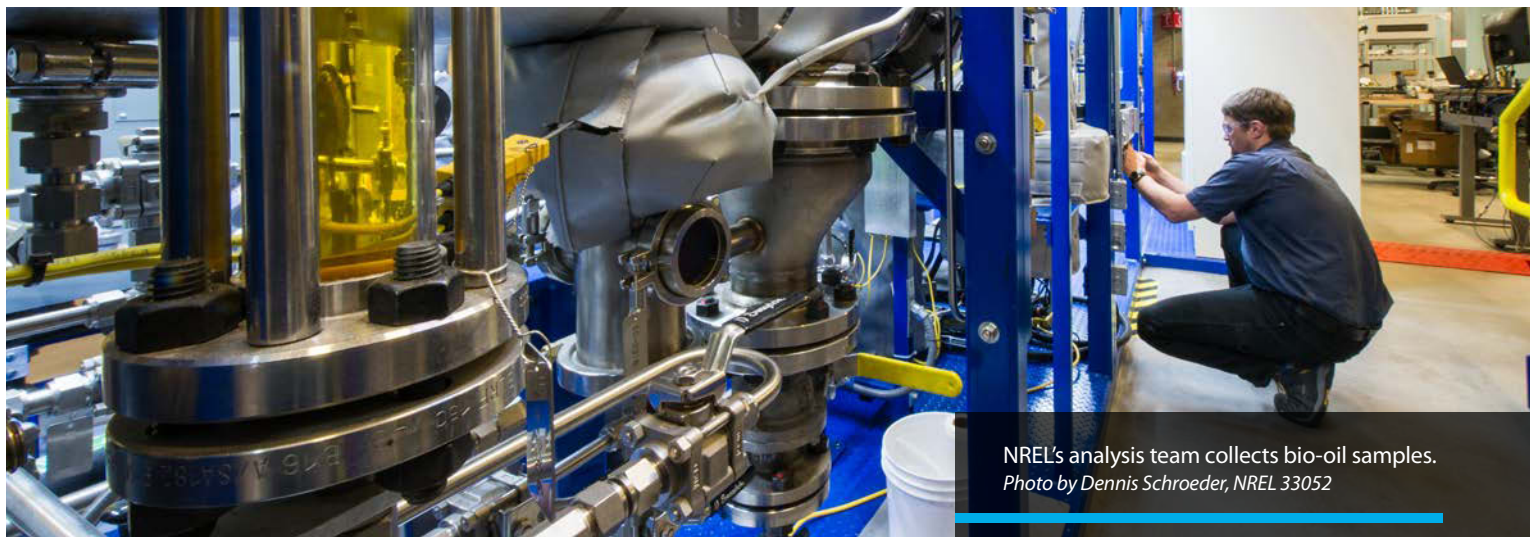
NREL established a standard method quantifying three different oxygenated functional groups: phenols, aliphatic alcohols, and carboxylic acids. Quantifying the concentration of these oxygenated functional groups helps guide the development of downstream upgrading and separations processes.

### METHOD VALIDATION

Methods are validated using round robin studies with at least five different laboratories. Inter-laboratory variability is assessed in the round robin, and this variability is critical to understanding the reliability of each method.

### CUSTOM METHOD DEVELOPMENT

NREL performs custom method development for partners who seek specific information on a bio-oil sample. By leveraging NREL's long history of working with bio-oils, our partners receive trustworthy information through our analytical methods with quantified variabilities.



NREL's analysis team collects bio-oil samples.  
Photo by Dennis Schroeder, NREL 33052

## RECENT SUCCESSES

NREL worked with ASTM International and the bio-oil community to develop the first ASTM standard to provide chemical information on bio-oils: *ASTM E3146: Standard Test Method for Determination of Carbonyls in Pyrolysis Bio-Oils by Potentiometric Titration*. ASTM E3146 is based on the carbonyl titration method developed in-house at NREL.

### PREDICTING UPGRADING PERFORMANCE

By working with researchers performing downstream upgrading of a fast pyrolysis bio-oil, NREL determined that bio-oils with  $>1.5$  mol carbonyl/gram would cause plugging during high-temperature hydroprocessing. Samples with a carbonyl content higher than this require further processing before being sent to the high-temperature hydroprocessing reactor. Therefore, NREL's carbonyl titration method is critical in determining safe hydrotreater operating conditions and preventing process downtime.

### CARBONYL TITRATION ENABLES NEW AGING TEST

Bio-oils become unstable with time and their properties change during long-term storage. The aging process increases viscosity, yet accelerated aging tests based on viscosity have proven unreliable due to high variability. Recently, NREL developed an accelerated aging test based on our carbonyl titration method. This test determines aging properties of a bio-oil, which can be used to determine how long a bio-oil can be stored and still be safely and effectively processed.

## Highlighted Publications

J. Ferrell, et al. "Standardization of Chemical Analytical Techniques for Pyrolysis Bio-Oil: History, Challenges, and Current Status of Methods." *Biofuels, Bioproducts & Biorefining*. DOI: 10.1002/bbb.166.

S. Black and J. Ferrell. "Determination of Carbonyl Groups in Pyrolysis Bio-Oils Using Potentiometric Titration: Review and Comparison of Methods." *Energy & Fuels*. DOI: 10.1021/acs.energyfuels.5b0251.

H. Ben and J. Ferrell. "In-Depth Investigation on Quantitative Characterization of Pyrolysis Oil by  $^{31}\text{P}$  NMR." *RSC Advances*. DOI: 10.1039/C5RA23939G.

## Find Out More

For more information and collaboration opportunities, contact: Jack Ferrell, [jack.ferrell@nrel.gov](mailto:jack.ferrell@nrel.gov)

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