U.S. DEPARTMENT OF ENERGY
Office of ENERGY EFFICIENCY & RENEWABLE ENERGY

Directed Funding Opportunity for Biomass Compositional Analysis

Informational Webinar

Beau Hoffman, Bioenergy Technologies Office (BETO)

Justin Sluiter, National Renewable Energy Laboratory (NREL)

May 11, 2021

Today's Speakers

Beau Hoffman

Technology Manager, BETO

Justin Sluiter

Researcher III, NREL

Webinar Housekeeping

- Attendees will be in listen-only mode
- Audio connection options:
  - Computer audio
  - Dial in through your phone (best connection)
- Technical difficulties? Contact us through the chat section, lower right of your screen
- Today's webinar will be recorded and posted to NREL's website: www.nrel.gov/bioenergy/biomass-compositional-analysis-dfo.html

Directed Funding Opportunity for Biomass Compositional Analysis Informational Webinar

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Justin Sluiter, NREL
May 11, 2021

EERE - Bioenergy Technologies Office

Vision — A thriving and sustainable bioeconomy fueled by innovative technologies

Mission — Developing transformative and revolutionary sustainable bioenergy and coproduct technologies for a prosperous nation

Strategic Goals — Develop industrially relevant technologies to enable domestically produced biofuels, biopower, and coproducts

Feedstock Technologies — Feedstock

Advanced Algal Systems — Algae

Conversion Technologies — Conversion

Systems Development and Integration — Systems

Data, Modeling, and Analysis — Data

Conversion Technologies Strategic Goal: develop efficient and economical biological and chemical technologies to convert biomass feedstocks into energy-dense liquid transportation fuels, such as renewable gasoline, diesel, and sustainable aviation fuel, as well as bioproducts, and chemical intermediates

Research and Development Funding areas:
Deconstruction and Synthesis
Bioprocessing R&D
Catalyst R&D
Co-Product R&D

Investing in the Bioeconomy

Biomass is the world's largest generator of renewable energy

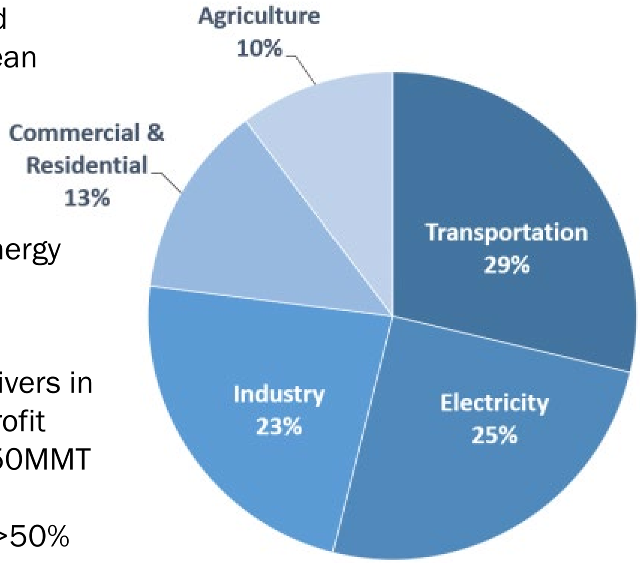
Biomass production can help fix carbon soil, increase use of non-productive lands, and improve ecosystems services, such as clean water and erosion control.

- Ag land emits >600MMT CO2 (2019)
- **Waste to Energy** Manure handling accounted for 82 MMT in 2019.
- **Renewables** for Irrigation and other energy needs.

Chemicals and Products are economic drivers in the biorefinery. >20% of oil use, > 45% profit

- Total GHG emission for chemicals >250MMT CO2 (2010)
- Bioproducts GHG reduction potential >50%
- Creates Biomass market demand
- Reduces fuels technology risk

Total U.S. Greenhouse Gas Emissions by Economic Sector in 2019



Demand for mobility in the US is **projected to grow** with population and economy:

- Light-duty vehicles: +20% by 2050
- Trucking: **+40%** by 2050
- Aviation: **+70%** by 2050

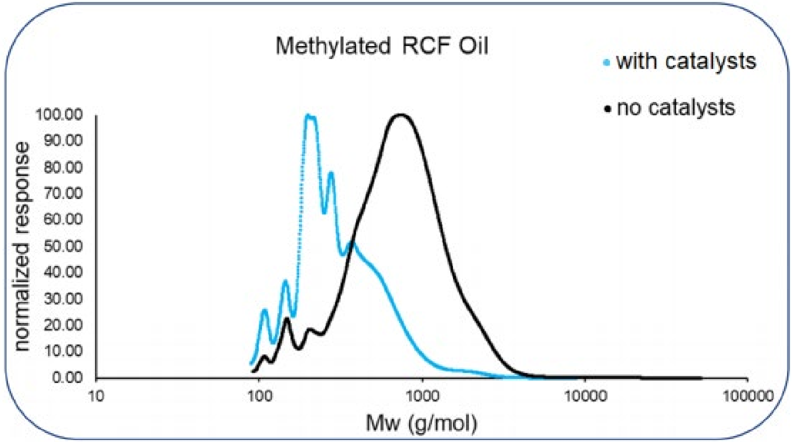
Energy need for "hard-to-electrify" heavy vehicles is projected to reach ~70 B gallon in 2050:

- Aviation: 36 B Gal
- Maritime/Rail: 11 B Gal
- Long-haul trucks: 21 B Gal

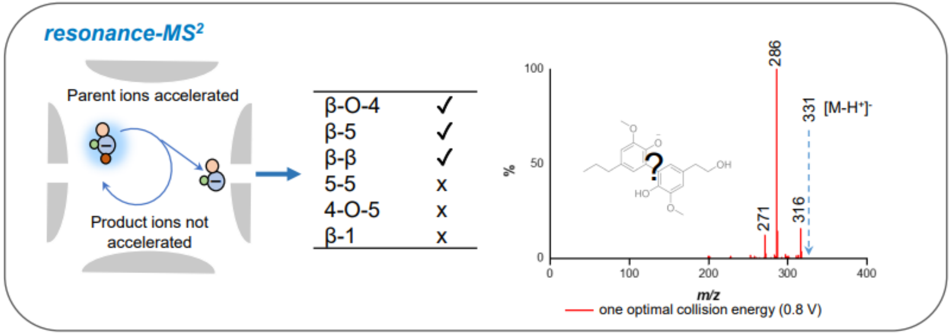
Inventory of U.S. Greenhouse Gas Emissions and Sinks: 1990–2019.

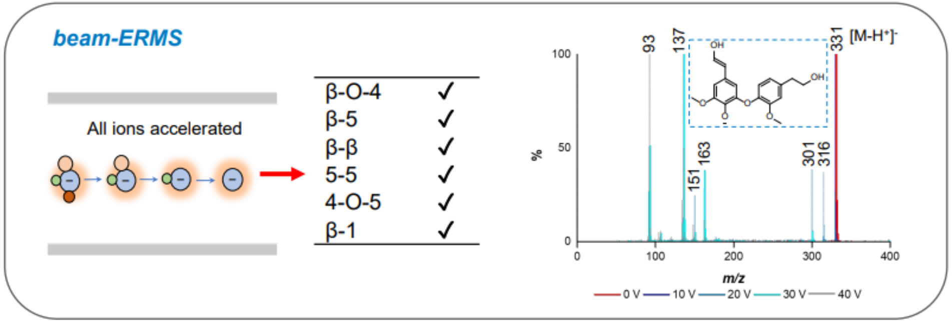
Urgent Analytical Needs – Lignocellulosic materials

- Lignin speciation
  & mass balance closure



K.P. Sullivan et al. in preparation





X. Dong, B.A. Black et al. in review at Green Chem

Urgent Analytical Needs - Carbohydrates

Direct determination of cellulosic glucan content in starch-containing samples

Justin B. Sluiter · Katie P. Michel · Bennett Addison · Yining Zeng · William Michener · Alexander L. Paterson · Frédéric A. Perras · Edward J. Wolfrum

Spike	Level (wt%)	N	Simple estimates			Mixed effect regression model		
			Mean (wt%)	SD	CV (%)	Mean (wt%)	SD	CV (%)
Biomass A								
No spike	0	18	1.95	0.13	6.5	1.9	0.04	2.1
Avicel cellulose	0.5	9	2.34	0.09	3.7	2.4	0.04	1.7
Avicel cellulose	1	5	3.03	0.09	3.1	2.9	0.04	1.5
Avicel cellulose	10	5	11.34	0.34	3.0	11.2	0.11	1.0
Resistant starch	0.5	9	2.02	0.11	5.7	2.0	0.06	3.0
Resistant starch	1	5	1.97	0.14	7.1	2.0	0.06	2.8
Resistant starch	10	5	2.22	0.07	3.1	2.2	0.04	1.8
Biomass B								
No spike	0	16	5.21	0.06	1.2	5.2	0.038	0.7
Avicel cellulose	0.5	9	5.63	0.17	3.0	5.7	0.039	0.7
Avicel Cellulose	1	5	6.32	0.17	2.7	6.1	0.040	0.7
Avicel cellulose	10	6	14.69	0.50	3.4	14.8	0.115	0.8
Resistant starch	0.5	9	5.17	0.10	1.9	5.2	0.058	1.1
Resistant starch	1	5	5.14	0.13	2.5	5.2	0.056	1.1
Resistant starch	10	5	5.21	0.05	1.0	5.2	0.039	0.8

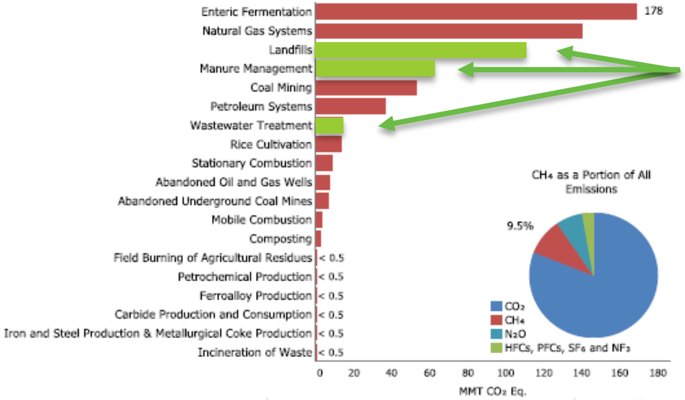
Exceptional precision

- The EPA released a guidance memo that requires a 10% CV for replicate analysis of cellulose.
  - at the time this was a challenging target
  - This could potentially require many replicates
- **The NREL method has measured CV's of $\leq$ 3.0%**

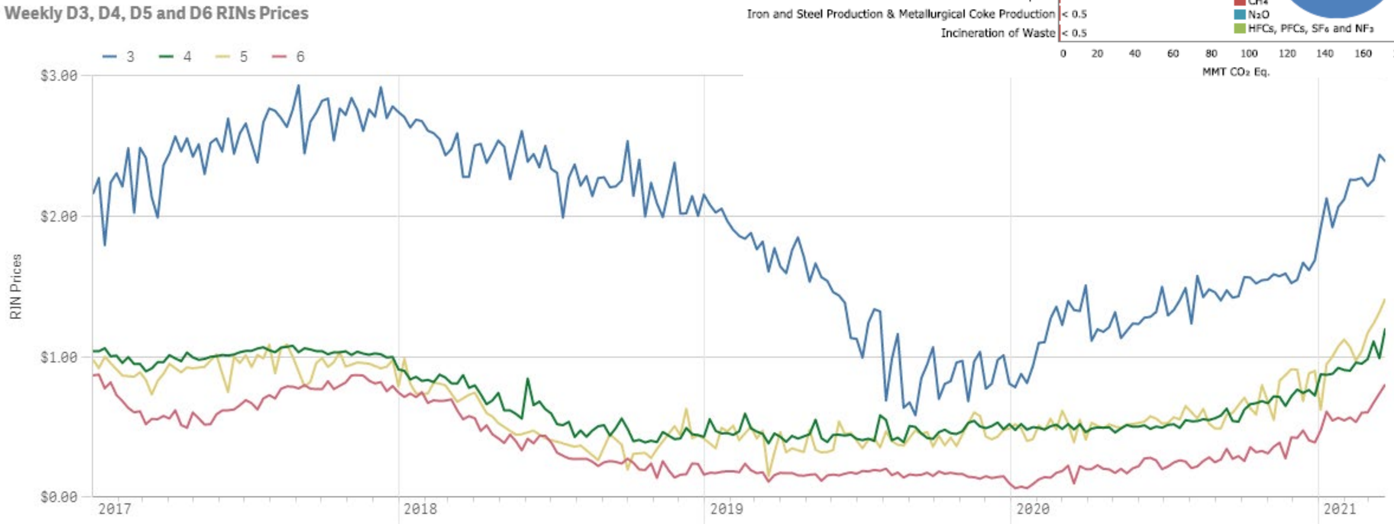
The low CV% allows for fewer replicates required to reach EPA targets, saving time and money.

Urgent Analytical Needs – Organic Waste

Organic Waste Speciation



>230 MMT/yr GHG emissions (CH_4, NO_x, CO_2)



$2.44/rin (~$31.68/mmbtu)

$1.32/RIN ($17.14/mmbtu)

Laboratory Capabilities

The Analytical Development and Support Project

The Analytical Directed Funding Opportunity will be added to an established project at NREL.

The Analytical Development and Support project at NREL is responsible for developing and maintaining analytical methods that support the biochemical conversion platform.

We are a team of analytical chemists with over 50 years of experience with carbohydrate chemistry.

The team collaborates with external stakeholders in the biofuels area, offering compositional analysis and method development for novel feeds or conversion products.

We are active with ASTM.

We are working with NIST and EPA to develop new reference materials.

Analytical Development and Support Team

Justin Sluiter

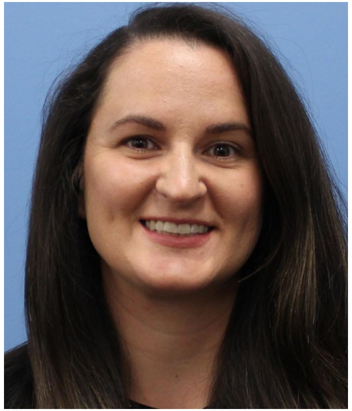
Justin's research interests include carbohydrate chemistry in biomass feedstocks and intermediate products. He leads the method development efforts both internally for the program and externally for industry stakeholders. His areas of interest are carbohydrate speciation in mixed carbohydrate streams, MSW, and wet waste characterization. Justin leads the Analytical Development and Support Project.

Ed Wolfrum

Ed's research interests include understanding how low-cost, rapid, non-destructive spectroscopic techniques combined with multivariate statistical analysis can be used for rapid biomass characterization. His key technical competencies include biomass compositional analysis using both conventional and rapid analysis methods, low-temperature biomass conversion, experimental design and exploratory data analysis, and multivariate statistics.

Darren Peterson

Darren's research interests are developing analytical methods to quantify the composition of various waste streams, such as brown grease. Darren is also developing methods to quantify carbohydrate utilization that undergo anaerobic digestion. He has extensive knowledge and is a valuable resource for many of NREL's Laboratory Analytical Procedures pertaining to biomass composition. He is continuously providing support for improving methods and making them less labor intensive.



Katie Michel

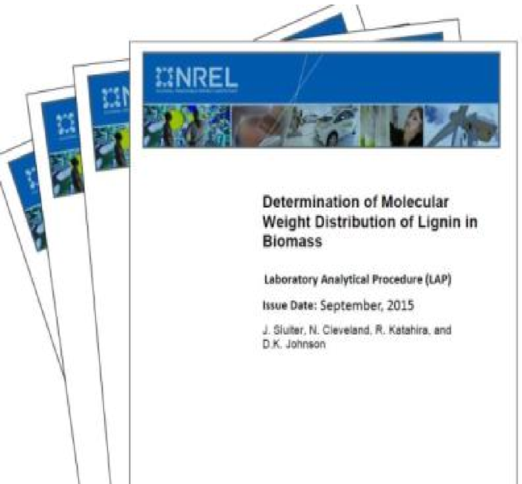
Katie came to NREL with over 12 years of experience in various technical laboratory settings. She brings knowledge of commercial analytical practices and expertise in HPLC method development. She assists with method development and optimization efforts. With expertise in data analysis and quality control, she has taken over the data management for the project.

Laboratory Analytical Procedures (LAPs)

NREL develops laboratory analytical procedures (LAPs) for standard biomass analysis. These procedures help scientists and analysts understand more about the chemical composition of raw biomass feedstocks and process intermediates for conversion to biofuels.

By combining the appropriate LAPs, the goal is to break the biomass sample down into constituents that sum to 100% by weight. Some of these constituents are individual components, such as individual carbohydrates, and some are groups of compounds, such as extractable material. However, the goal of these analyses is to characterize all of the material in the sample

TITLE	CITED BY	YEAR
Determination of structural carbohydrates and lignin in biomass A Sluiter, B Hames, R Ruiz, C Scarlata, J Sluiter, D Templeton, D Crocker Laboratory analytical procedure 1617 (1), 1-16	6107 *	2008
Determination of extractives in biomass	1051 *	2005



Non-structural Material: Water solubles including Glucose, Sucrose, Fructose; ethanol solubles

Structural Material: Glucans, Xylans, Galactan, Arabinan, Mannan, Lignin

Inorganics, protein, and total solids content are also quantified

Procedure have been applied to woody and herbaceous crops, grains, animal and food wastes, MSW, and various conversion treatment products of the same.

Compositional Analysis Systems

- Cellulosic Glucan Content in Starch Containing Feedstocks
- Preparation of Samples for Compositional Analysis
- Total Solids in Biomass and Total Dissolved Solids in Liquid Process Samples
- Ash in Biomass
- Protein Content in Biomass
- Extractives in Biomass
- Structural Carbohydrates and Lignin in Biomass
- Sugars, Byproducts, and Degradation Products in Liquid Fraction Process Samples
- Insoluble Solids in Pretreated Biomass Material
- *cis,cis*- and *cis,trans*-Muconic Acid from Biological Conversion

Characterization Equipment	
System Type	Systems Available

Thermo Accelerated Solvent Extractor (ASE 350)	• Characterization of non-structural components.
	    • Waxes
	    • Fats
	• Small scale acid treatment

Agilent 1100 or 1200 HPLC with Refractive index or UV detection	• Carbohydrate quantification • Organic acids quantification
NanoDrop™ 8000 Spectrophotometer	• Quantification of soluble lignin
Fully equipped analytical laboratory: Autoclaves, balances, Furnace, Solids analysis, Soxhlet, mills, sieves, filtration, CHN analysis	

Relevant Publications:

Sluiter, A.; Sluiter, J.; Wolfrum, E.J. (2013). “Methods for Biomass Compositional Analysis, in Catalysis for the Conversion of Biomass and Its Derivatives.” M.a.A.D. Behrens, Ed. Berlin, Germany: Max Planck Research Library for the History and Development of Knowledge, Proceedings 2 pp. 213-254.

Sluiter, J.B., et al. (2010). “Compositional Analysis of Lignocellulosic Feedstocks. 1. Review and Description of Methods.” J. Ag. Food Chem. (58:16); pp. 9043-9053.

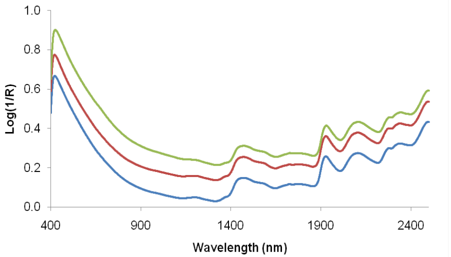
Templeton, D.W., et al. (2010). “Compositional Analysis of Lignocellulosic Feedstocks. 2. Method Uncertainties.” J. Ag. Food Chem. (58:16); pp. 9054-9062.

Wolfrum, E., et al. (2009). “Correlating Detergent Fiber Analysis and Dietary Fiber Analysis Data for Corn Stover Collected by NIRS.” Cellulose (16:4); pp. 577-585.

Sluiter, J.B., Michel, K.P., Addison, B. et al. Direct determination of cellulosic glucan content in starch-containing samples. Cellulose 28, 1989–2002 (2021).

Predictive Modeling Systems

System	Specification	Software Package
Metrohm XDS	• Dispersive NIR • 400nm to 2500nm • multiple scanning modes, including 20-vial high-throughput tray	VISION™ operating software
Thermo Antaris II	• FT-NIR • 3800cm-1 to 12000cm-1 • multiple scanning modes, including 40-sample high-throughput carousel	OMNIC™, RESULT™ operating software packages



Relevant Publications:

"A Performance Comparison of Low-Cost Near-Infrared (NIR) Spectrometers to a Conventional Laboratory Spectrometer for Rapid Biomass Compositional Analysis", Edward J. Wolfrum, Courtney Payne, Alexa Schwartz, Joshua Jacobs, Robert W. Kressin, Bioenergy Research 13(4):1121-1129 (2020).

"Quantitative trait loci for cell wall composition traits measured using near-infrared spectroscopy in the model C4 perennial grass *Panicum hallii*", E. Milano, C. Payne, E. Wolfrum, J. Lovell, J. Jenkins, J. Schmutz, T. Juenger. *Biotechnology for Biofuels 11:25 (2018).*
"High throughput screening technologies in biomass characterization", Stephen R. Decker, Renee M. Happs, Anne E. Harman-Ware, Edward J. Wolfrum, Daniel Jacobson, Deborah Weighill, Piet C. Jones, Gerald A. Tuskan, David Kainer, Miguel Rodriguez, Gbekeloluwa Oguntimein. *Frontiers in Energy Research 6:120 (2018).*
"Evaluation of fifteen cultivars of cool-season perennial grasses as biofuel feedstocks using NIR/PLS", C. Payne, E. Wolfrum, N. Nagle, J. Brummer, N. Hanson. *Agronomy Journal 109:1923-1934 (2017).*
"Rapid analysis of composition and reactivity in cellulosic biomass feedstocks with near-infrared spectroscopy". Courtney E. Payne and Edward J Wolfrum, *Biotechnology for Biofuels 8:43(2015). doi:10.1186/s13068-015-0222-2*
"Multivariate Calibration Models for Sorghum Composition using Near-Infrared Spectroscopy", E. Wolfrum and C. Payne (NREL), T. Stefaniak and W. Rooney, N. Dighe, B. Bean, J. Dahlberg, "Near infrared calibration models for pretreated corn stover slurry solids, isolated and in-situ", Amie Sluiter and Ed Wolfrum. *J. Near-Infrared Spectroscopy 21(4):249-257 (2013).* "Improved multivariate calibration models for corn stover feedstock and dilute-acid pretreated corn stover", E. Wolfrum, A. Sluiter. *Cellulose 16(5):567-576 (2009).*

Advanced Analytical Systems for Validation

Chromatographic Equipment	
System Type	Systems Available
Qualitative Mass Spec Systems	• Ion /mobility Spec Quadrupole Time of Flight Mass Spec • Ion Trap Mass Spec
Quantitative Mass Spec Systems	• Single Quadrupole – Mass Spec • Triple Quadrupole Mass Spec
Quantitative LC Systems	• UPLC/HPLC • HPLC Agilent 1260 • Ion chromatography with pulsed amperometric detection
Qualitative/Quantitative GC Systems	• GC-MS • GC-FIC

Magnetic Resonance Equipment Specifications

System	Capabilities
600 MHz, 14.7 Tesla Bruker Avance III NMR Spectrometer	Liquid and solid state capabilities Bruker SampleJet for high-throughput analysis High sensitivity liquid-state Bruker CryoProbe High resolution MAS probe for semi-solid samples MAS spinning speeds up to 35 kHz
400 MHz, 9.4 Tesla Bruker Avance III HD NanoBay NMR Spectrometer	Dedicated liquids system Bruker SampleCase autosampler Bruker Prodigy CryoProbe for increased sensitivity Routine ^{1}H, ^{13}C, ^{31}P, and ^{19}F Temperatures studies from -40°C to 80°C
200 MHz, 4.7 Tesla Bruker Avance III HD NMR Spectrometer	Ideal for solid-state ^{13}C NMR studies 10 mm liquids probe for rapid ^{13}C analysis

Topic Area 1:
Characterization of Mixed Carbohydrate Streams

This area of interest seeks to improve understanding of mixed carbohydrate usage during conversion to biofuels, bioproducts, or biopower by developing, improving, or assessing analytical methods for speciation and quantifications of poorly quantified analytes.

For the purposes of this DFO, mixed carbohydrate streams are streams that contain both cellulosic and non-cellulosic carbohydrates, for example streams containing both cellulose and starch.

The objective for this topic area is to produce a new published procedure that will aid in characterization of feed and process streams that will enable greater understanding of conversion technologies.

Topic Area 2: Rapid Characterization Methods for Mixed Carbohydrate and Lignocellulosic Streams

This area of interest seeks proposals to advance development of rapid measurement techniques, such as spectroscopic methods, that enable at-line or on-line stream characterization for use by biomass carbohydrate, mixed carbohydrate, lignocellulosic, or waste-to-energy conversion facilities.

The objective for this topic area is to produce a rapid characterization method based on largely existing analytical methodology that will enable on-line or at-line characterization.

Some development of primary analytical methods may be within the scope of this topic area, but he majority of the focus should be on development of spectroscopic methodology.

Topic Area 3: Analytical Methods for Characterization of Wet Organic Wastes

This area of interest seeks to develop standardized analytical methods to support cost-advantaged feedstocks, including (but not limited to) feed streams for anaerobic digestion that improve understanding of components present in the streams.

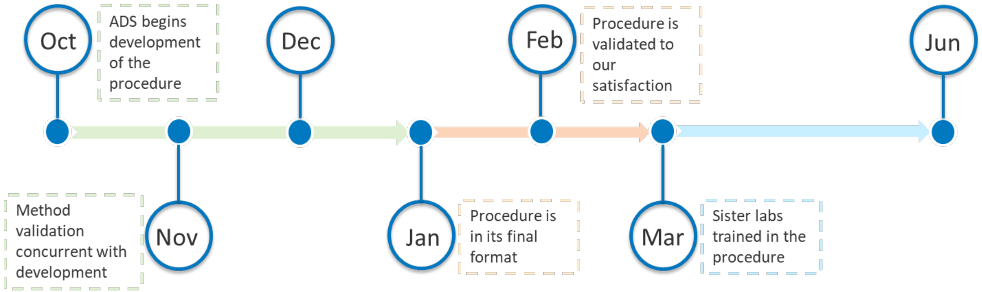
For the purposes of this DFO, wet organic wastes are waste materials containing at least 40% water by weight. Typical wet wastes include (but are not limited to) manure, biosolids from water treatment facilities, and food waste.

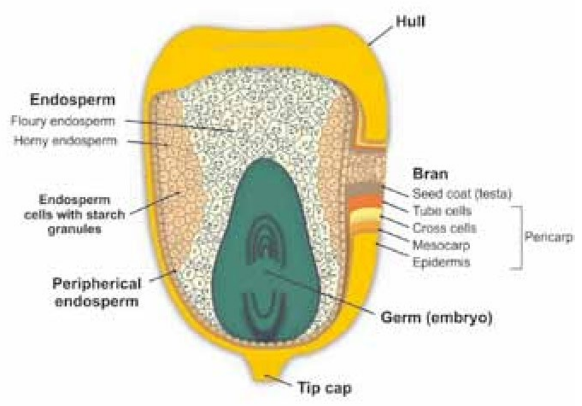
The objective for this topic area is to produce a new published procedure that will aid in characterization of feed and process streams that will enable greater understanding of conversion technologies.

An Example:
Insitu Cellulose Conversion Gen 1 Ethanol

Recently the EPA expressed a desire for a publicly transparent analytical method that can measure small quantities of cellulose in samples with significant starch present. NREL was asked to develop a novel method that met the precision and accuracy requirements of the EPA and that would be publicly transparent for stakeholder evaluation.

Involving other national laboratories, we performed a round robin to validate our quantified precision and accuracy.





Corn kernel composition.
Adapted from Eckhoff and Watson (2009)

The ADS group developed this new method in under a year.

An Example:
Insitu Cellulose Conversion Gen 1 Ethanol

The method met the objectives for precisions specified by the EPA

ADS used advanced analytical techniques to validate the method and ensure accuracy.

- NMR was used to ensure no residual starch present in the cellulose fraction.
- LC/MS was used to ensure no loss of cellulose during the starch removal.

The method is now published, and the method is available for download as a LAP.

Cellulose (2021) 28:1989–2002
https://doi.org/10.1007/s10570-020-03652-2

ORIGINAL RESEARCH

Direct determination of cellulosic glucan content in starch-containing samples

Justin B. Sluiter · Katie P. Michel · Bennett Addison · Yining Zeng · William Michener · Alexander L. Paterson · Frédéric A. Perras · Edward J. Wolfrum

Received: 30 June 2020 / Accepted: 15 December 2020 / Published online: 15 January 2021

Determination of Cellulosic Glucan Content in Starch Containing Feedstocks

Laboratory Analytical Procedure (LAP)

Issue Date: February 26, 2021

Timeline and Process for DFO

Beau Hoffman

Biomass Compositional Analysis DFO

- **Goal**: The goal of the Biomass Compositional Analysis DFO is to develop new analytical and on-line / at-line measurement systems for industrial and academic partners
  - Leverage expertise in biomass characterization
  - Publish third-party credible analyses

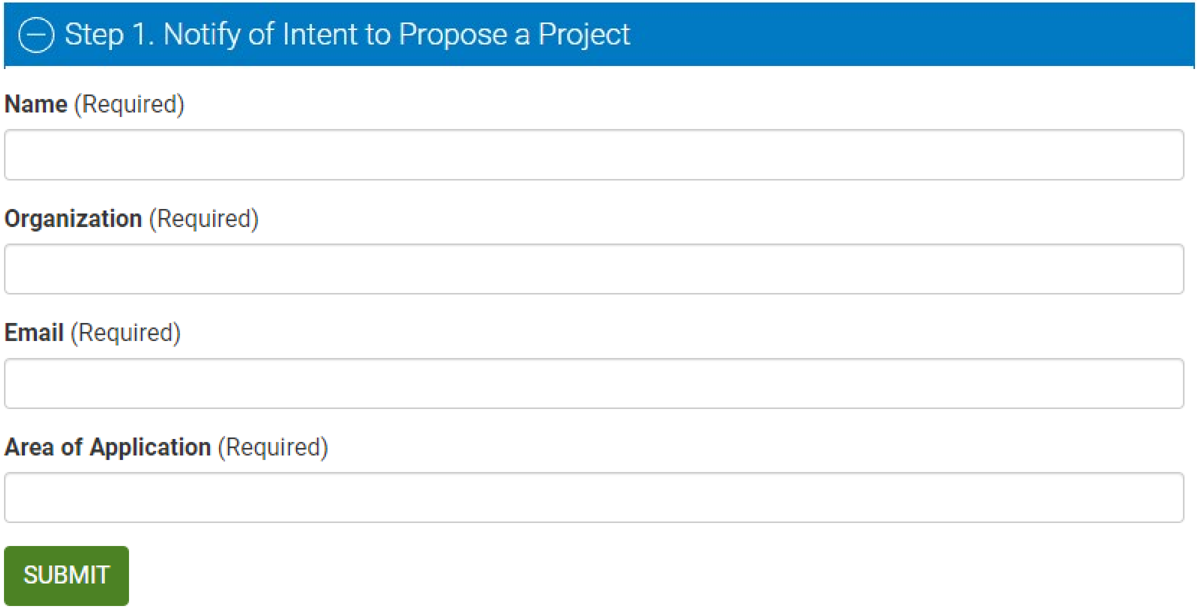
- **Eligibility**: For-profit and academic institutions. Foreign entities are eligible to apply but must receive approval from DOE if selected
- **Cost:** All federal funds will be spent by the National Renewable Energy Laboratory. No funds will "pass-through" to partner organizations. Partner organizations are required to contribute > 20% cost-share
- **Award Size:** Proposals should be written to $150k - $500k of Federal funds. A total of $1.5M is available for the entire program.

Timeline and Process

- May 11th – Informational Webinar
- May 24 – 28 – Applicant presentations with NREL
- June 11th– Notice of Intent Deadline
- June 18th– Proposal Submission Deadline
- June/July– Project Proposal Review
  - 3rd party reviewers will assess the merit and potential impact of the project
- Late July – Announcement of Selections
- September –Anticipated project kickoffs

Notice of Intent

A notice of intent is required by June 11th to understand interest in the program



Application Template

Technical Approach (2 pages)
Scope of Work (3-4 pages)
Impact (2-3 pages)
Rationale for gov't funding (1 page)

Maximum budget is $500k of Federal funds

Please use the template provided on the DFO webpage

Directed Funding Opportunity for Biomass Compositional Analysis

The U.S. Department of Energy (DOE) Bioenergy Technologies Office (BETO) is offering directed funding assistance for industry and universities interested in leveraging NREL's unique biomass compositional analysis capabilities.

⊕ Step 1. Notify of Intent to Propose a Project

⊕ Step 2. Meet with NREL Analysts

⊖ Step 3. Develop White Paper Proposal

Use the Biomass Analysis Technologies—Analytical Development Support DFO White Paper Template to ...elop your white paper proposal. Anticipated period of performance for all work should be less than 24 ...onths.

Proposal Review Factors

3rd Party Reviewers are being chosen to independently evaluate these request proposals. All reviewers will sign a conflict of interest/non-disclosure agreement

Reviewers will evaluate each proposal on the following criteria:

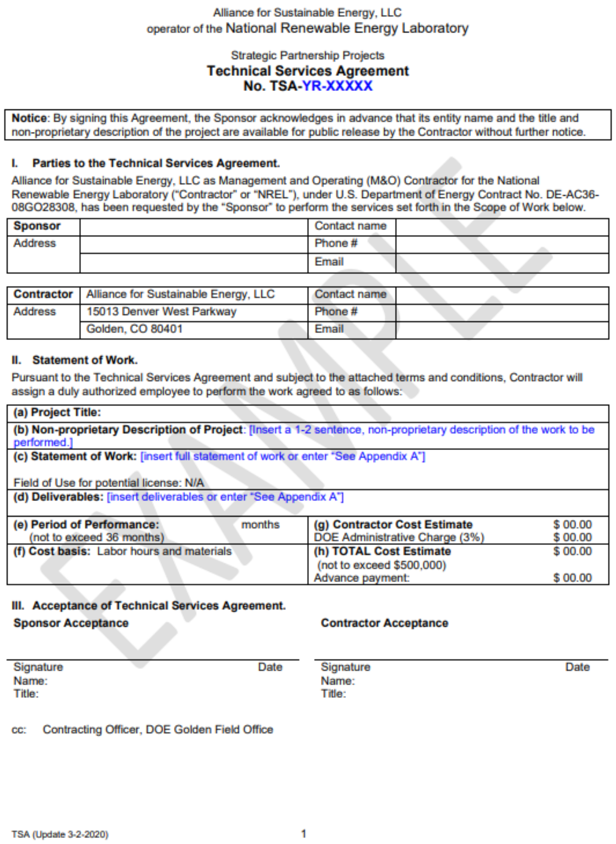
Technical approach: research plan, technical challenges addressed, NREL capabilities leveraged, milestones, proposed budget, and schedule	50%
Potential impact: targeting BETO goals, addressing technical barriers, and market impact on the biofuels and bioproducts industry	40%
Appropriateness of government funding, key personnel, and resources	10%

Contractual Info: Technical Service Agreements

Applicants are strongly encouraged to review the example TSA posted on the DFO website.

Terms are final to expedite the initiation of projects once projects are selected and announced

IP is not expected to be generated during this program: methods developed are intended to be published as laboratory analytical procedures for the biomass industry/research community to benefit from



Alliance for Sustainable Energy, LLC
operator of the National Renewable Energy Laboratory

Strategic Partnership Projects
Technical Services Agreement
No. TSA-YR-XXXXX

Notice: By signing this Agreement, the Sponsor acknowledges in advance that its entity name and the title and non-proprietary description of the project are available for public release by the Contractor without further notice.

I. Parties to the Technical Services Agreement.

Alliance for Sustainable Energy, LLC as Management and Operating (M&O) Contractor for the National Renewable Energy Laboratory ("Contractor" or "NREL"), under U.S. Department of Energy Contract No. DE-AC36-08GO28308, has been requested by the "Sponsor" to perform the services set forth in the Scope of Work below.

Sponsor		Contact name	
Address		Phone #	
		Email	

Contractor	Alliance for Sustainable Energy, LLC	Contact name	
Address	15013 Denver West Parkway	Phone #	
	Golden, CO 80401	Email	

II. Statement of Work.

Pursuant to the Technical Services Agreement and subject to the attached terms and conditions, Contractor will assign a duly authorized employee to perform the work agreed to as follows:

(a) Project Title:
(b) Non-proprietary Description of Project: [Insert a 1-2 sentence, non-proprietary description of the work to be performed.]
(c) Statement of Work: [insert full statement of work or enter "See Appendix A"] Field of Use for potential license: N/A
(d) Deliverables: [insert deliverables or enter "See Appendix A"]

(e) Period of Performance: (not to exceed 36 months)	months	**(g) Contractor Cost Estimate** DOE Administrative Charge (3%)	\$ 00.00 \$ 00.00
(f) Cost basis: Labor hours and materials		**(h) TOTAL Cost Estimate** (not to exceed \$500,000) Advance payment:	\$ 00.00 \$ 00.00

III. Acceptance of Technical Services Agreement.

Sponsor Acceptance	**Contractor Acceptance**
Signature Date Name: Title:	Signature Date Name: Title:

cc: Contracting Officer, DOE Golden Field Office

TSA (Update 3-2-2020) 1

FAQs

- **Will these slides be posted?**

  These slides and a recording of the webinar will be posted on the website
- **Will funding be available to companies/universities?**

  All federal funds under this program will be spent by researchers at the National Renewable Energy Laboratory
- **Are other DOE national laboratories allowed to apply?**

  No, this opportunity is limited to organizations external to the DOE National Laboratory complex
- **What is cost share?**

  Cost share principles are available in 2 CFR 200.306. In-kind cost share (such as technical consulting/expertise, or use of equipment) is allowed as is cash cost share

FAQs

- **How is 20% cost share calculated?**

  20% cost share is calculated based on the <u>total project cost</u> (not just the federal share). For example:

  A project is requesting $250k of Federal support, a minimum cost share of $62,500 would be required. $62,500 is 20.00% of $312,500.

  (Tip: Federal funds/0.8 = total project cost. Total project cost – Federal funds = minimum cost share)

- **Can I submit multiple proposals?**

  Yes, provided the requests are unique and distinct?

- **What if I have other questions?**

  Please visit the website to view our current list of FAQs. If your question has not been answered, please submit them to Analytical.DFO@nrel.gov

Thank you!

www.nrel.gov

NREL
Transforming ENERGY

Questions?

Beau Hoffman

beau.hoffman@ee.doe.gov

Justin Sluiter

justin.sluiter@nrel.gov

More about this DFO: https://www.nrel.gov/bioenergy/biomass-compositional-analysis-dfo.html

DFO Contact: analytical.DFO@nrel.gov

U.S. DEPARTMENT OF ENERGY | Office of ENERGY EFFICIENCY & RENEWABLE ENERGY

BIOENERGY TECHNOLOGIES OFFICE