



Production of 1,000 Gallons of Biojet in the NARA Consortium

R. J. Wooley¹, M. Wolcott², R. Cavalieri², J. Y. Zhu³,
R. Gleisner³, A. C. Hawkins⁴, J. Ley⁴, K. Evans⁴,
G. Johnston⁴, J. Gao⁵, T. Spink⁶, G. Marrs⁷, J. Sessions⁸

**NARA Final Meeting
Arlington, VA
November 17, 2016**

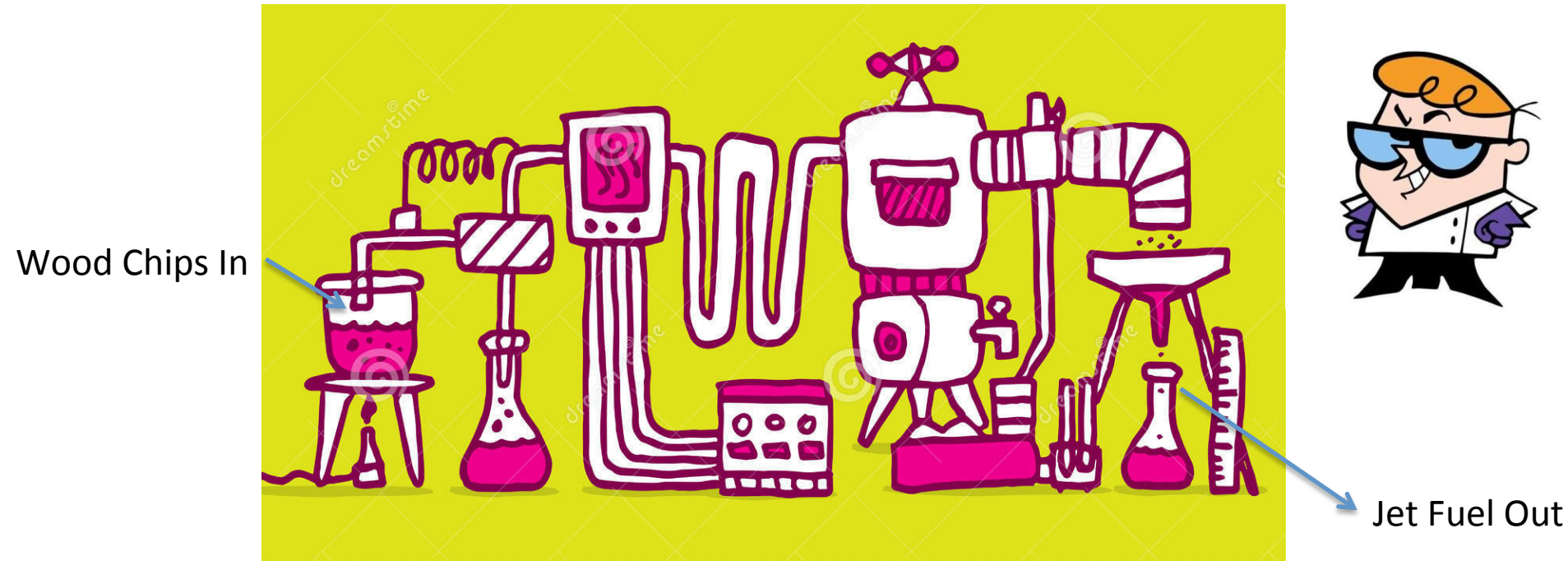
¹Biomass *ad infinitum*, LLC, ²Washington State University, ³Forest Products Laboratory,
⁴Gevo, Inc., ⁵Weyerhaeuser, Inc., ⁶TSI, Inc., ⁷Gevan Marrs, LLC, ⁸Oregon State University

Northwest Advanced Renewables Alliance



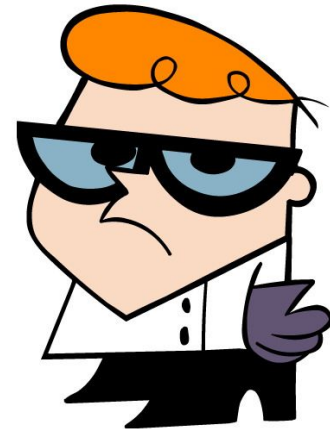
The Challenge!

- You have process in the lab/pilot plant

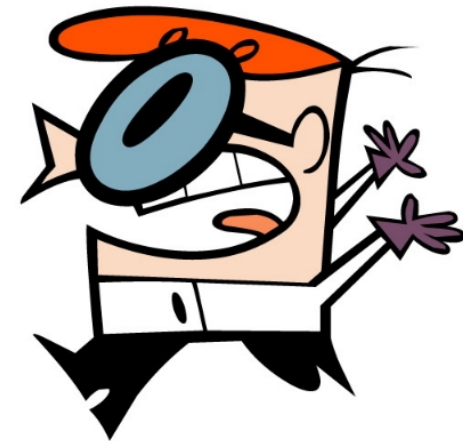


- You're able to validate the process & develop scale-up data
- You're able to supply quantities for analytical testing

- Then you realize that lab quantities are only going to get you so far



- You need enough fuel product to demonstrate a commercial flight



Produce 1,000 gallons of jet fuel using the feedstock and process identified and researched by the USDA funded NARA project.

1. Utilize Key aspects from the NARA project in the production:
 - Feedstock: Softwood forest residues, primarily Douglas-fir and hemlock
 - Pretreatment: A mild bisulfite variant of the SPORL process as developed by USDA/FPL and Catchlight Energy
 - Enzymatic Saccharification: Utilizing commercial enzymes from Novozymes and as utilized by USDA/FPL and Gevo on this pretreated material
 - Isobutanol Production: Via fermentation using Gevo patented organisms and fermentation protocols
 - Jet Fuel Conversion: Via Gevo process

1,000 Gallons of BioJet Fuel – Task Objective (cont.)

NARA – USDA Update, Washington, DC
November 17, 2016

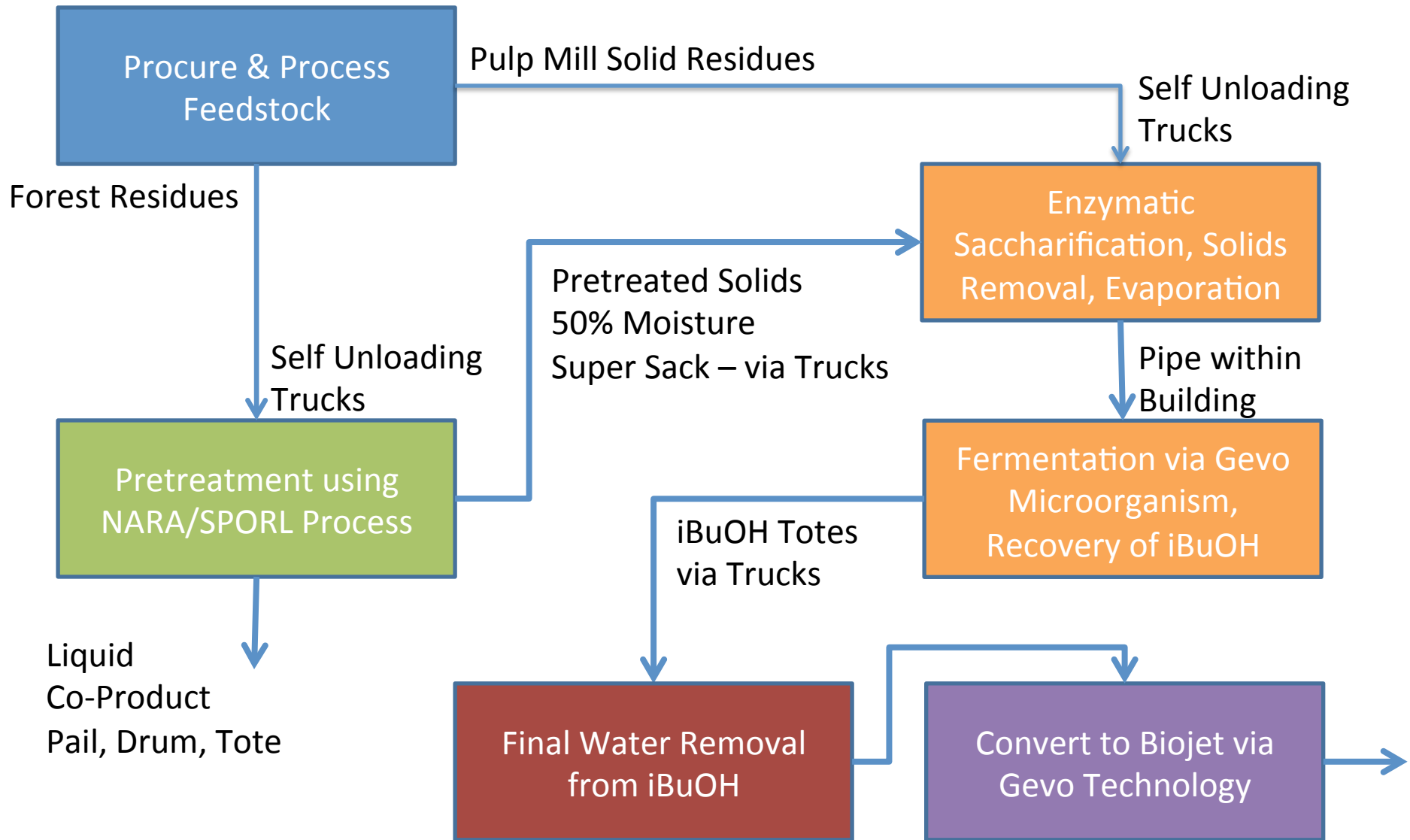
2. Efforts will be made to accommodate the production of representative co-products
3. Cost and availability of suitable demonstration scale equipment will dominate
4. Efforts will be made to determine representative or scalable yields as opportunities present themselves
5. An overall optimized yield from wood to jet fuel is not expected

Determine Scale-up Requirements

- Identify & Investigate Potential Tolling Partners
- Determine additional testing needed by FPL and Gevo to scale to a continuous system that may require different process conditions than previously studied
- Discuss potential need for pre-trials with specific tolling equipment
- Requirements and procedures for shipping of intermediate materials – Will depend on toller capabilities and locations
 - Low pH of hydrolyzates
 - Moisture content of solids
 - Bio-stability of sugar containing materials
- Negotiate Contracts with Each Selected Partner

Overall Process Flow

NARA – USDA Update, Washington, DC
November 17, 2016



Potential Tolling Partners Evaluated

NARA – USDA Update, Washington, DC
November 17, 2016

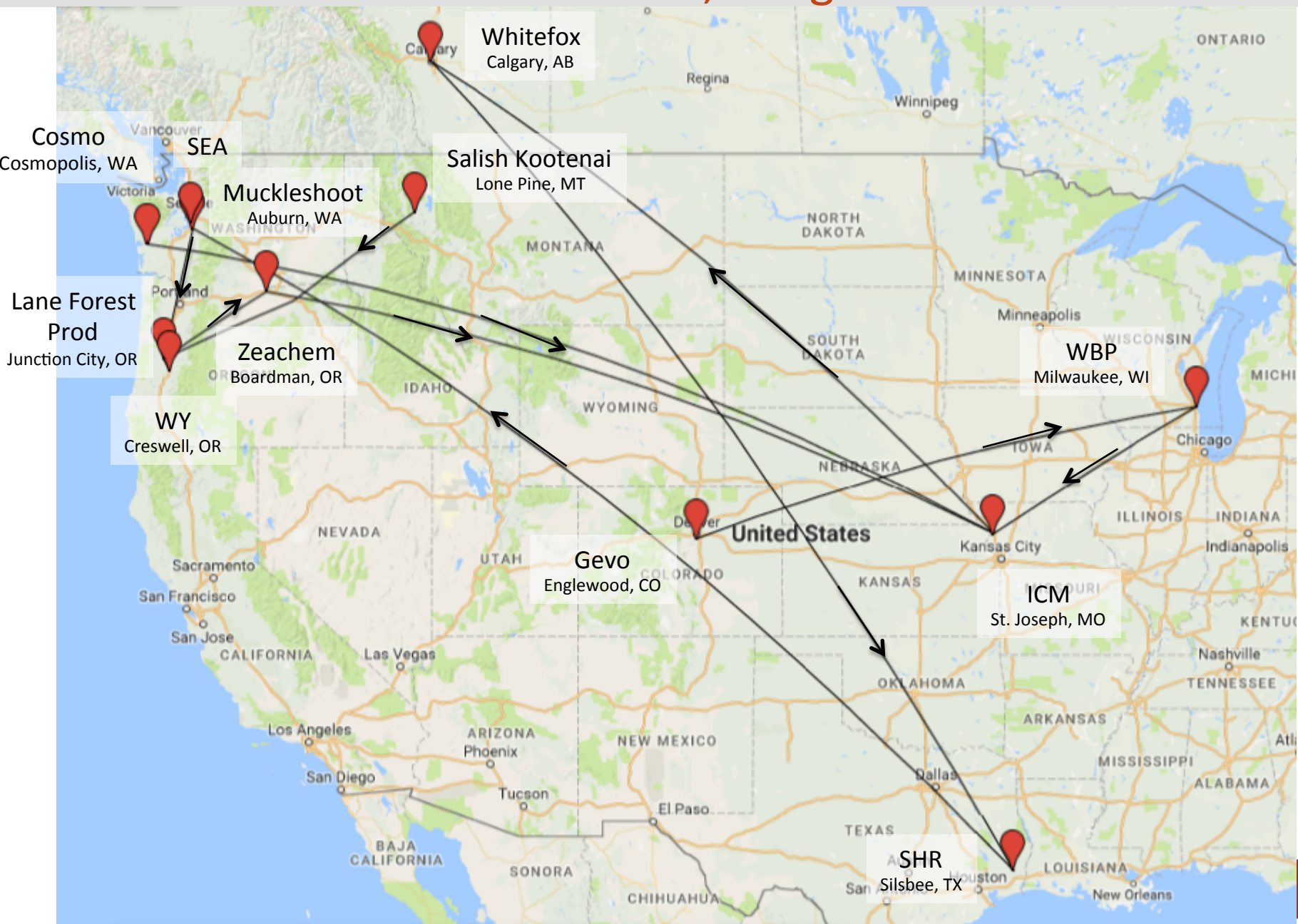
- Pretreatment
 - Andritz Pilot Facility, Springfield, OH
 - American Process (API) Pilot Plant, Thomaston, GA
 - Zechem Development Plant, Boardman, OR
 - Cosmo Specialty Fiber, Cosmopolis, WA
 - Forest Products Lab (FPL), Madison, WI
 - ICM Corn & Cellulose Pilot Plant, St. Joseph, MO
 - NREL Biomass Pilot Plant, Golden, CO
 - University of Florida Pilot Plant, Perry, FL
- Enzymatic Saccharification, Fermentation, Purification
 - American Process (API) Pilot Plant, Thomason, GA
 - ICM, St. Joseph, MO
 - NREL Biomass Pilot Plant, Golden, CO
- iBuOH Conversion to Jet
 - South Hampton Resources, Silsbee, TX



Northwest Advanced Renewables Alliance

Locations Involved to Produce 1,000 gallons

NARA – USDA Update, Washington, DC
November 17, 2016





Feedstock Collection

- Weyerhaeuser Creswell, Oregon
- CSTK Lands near Lone Pine, Montana
- Muckleshoot Lands near Auburn, Washington

Feedstock Processing

- Lane Forest Products, Junction City, Oregon
- Forest Concepts, Auburn, Washington

Feedstock – Modified to fit Specific Toller


NARA – USDA Update, Washington, DC
November 17, 2016

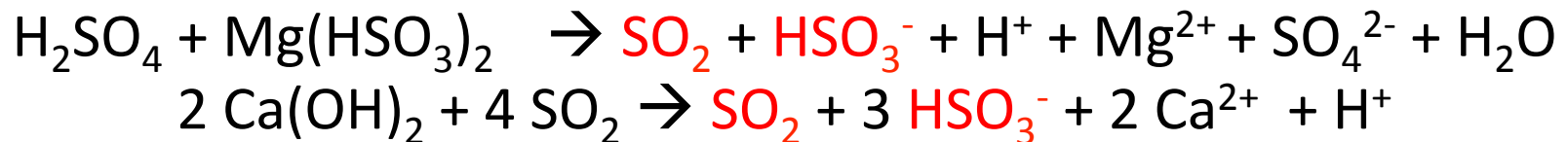
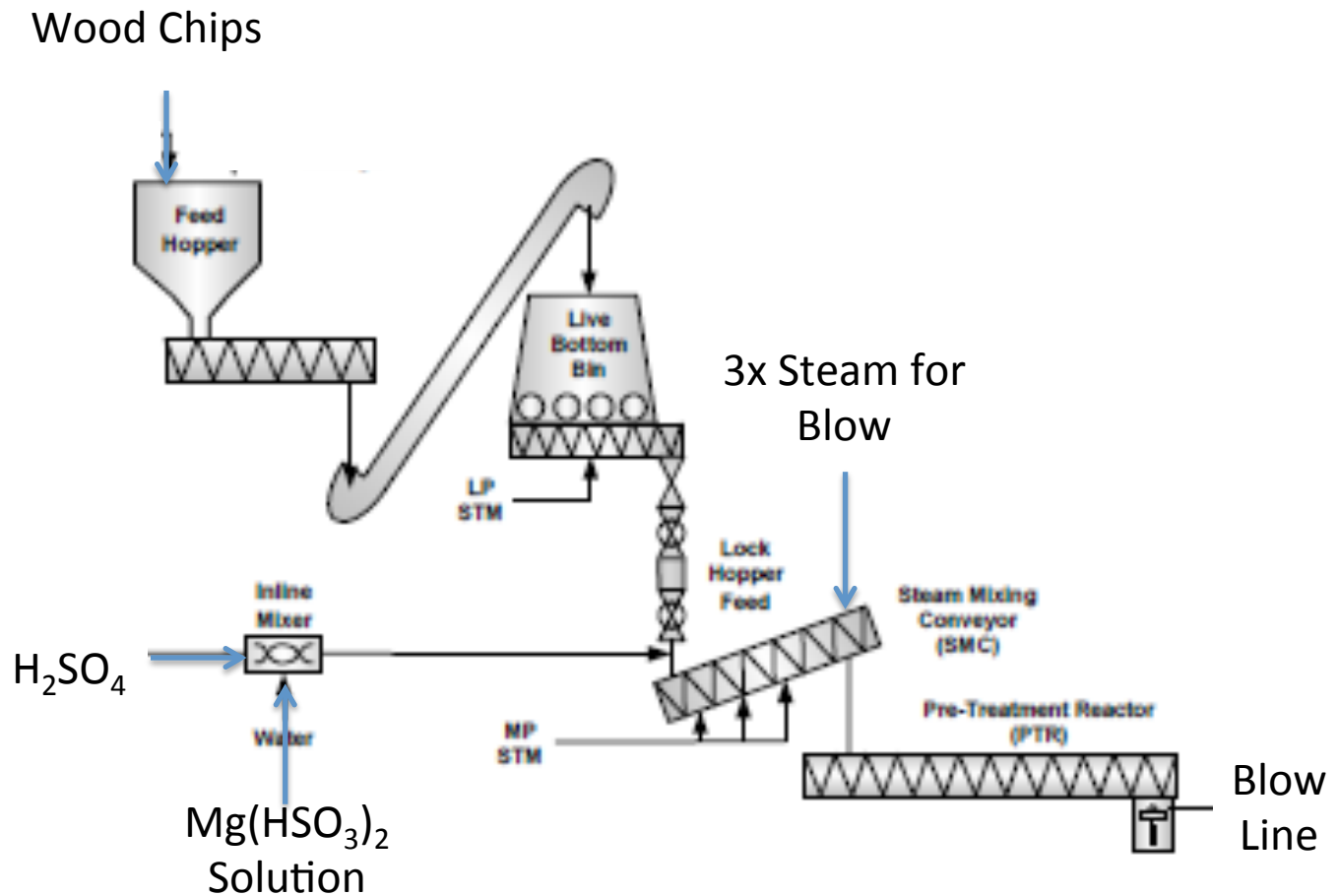


As Received at Lane Forest Products

Material Used at Zeachem

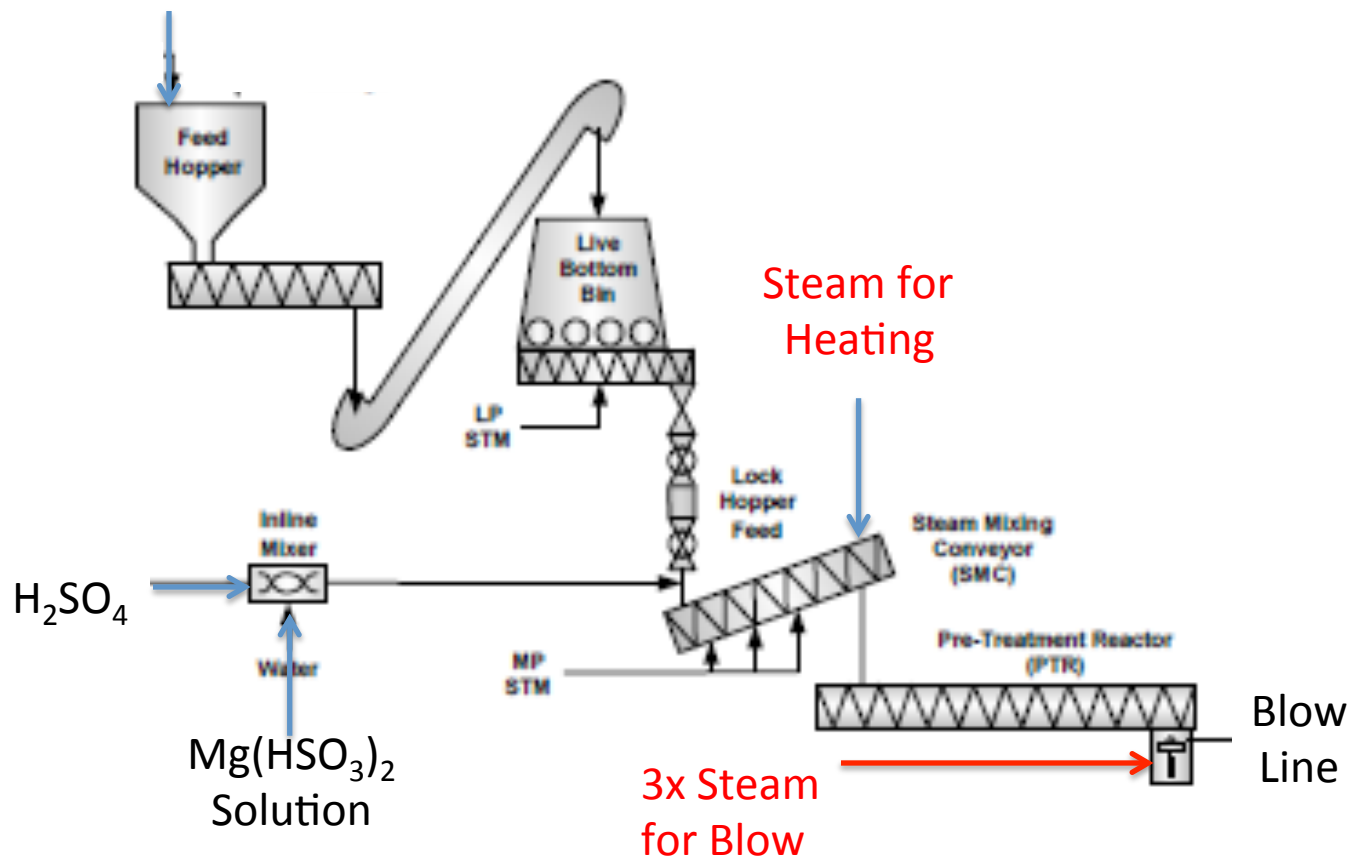


- 
- Zeachem – Boardman OR
 - Good Experience with pretreatment of wood for fuels and chemicals
 - Nominal 10 BDT/day
 - No SO₂ Handling Experience – Designs & Procedural Modifications
 - Andritz Designed and Supplied Equipment
 - Trial at Andritz Pilot (Springfield, OH)
 - Budget Quote for Andritz Commercial Plant for NARA
 - Refiner and High Pressure Blow will insure small particle size
 - Filter Press can produce 50% Solids, quite suitable for non-Hazmat Shipping
 - Highly motivated team at Zeachem

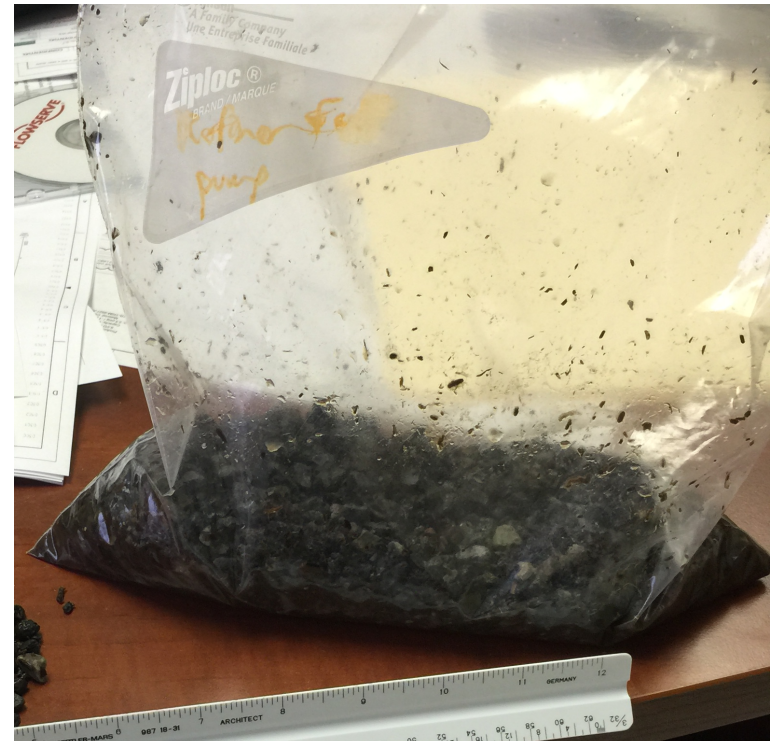


Zechem Solving Problem of Low SO₂ Partial Pressure

Wood Chips

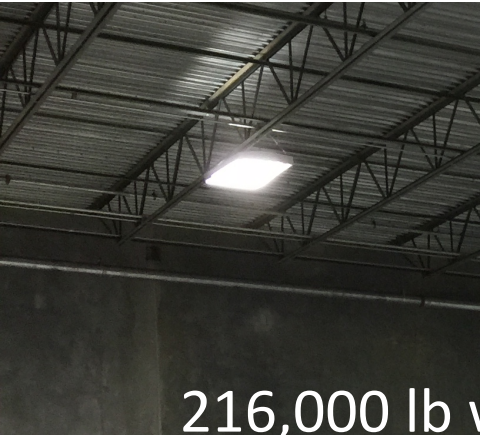


- Rocks in the Feedstock!



Zeachem 52 BDT of Pretreated Solids Shipped to ICM

NARA – USDA Update, Washington, DC
November 17, 2016



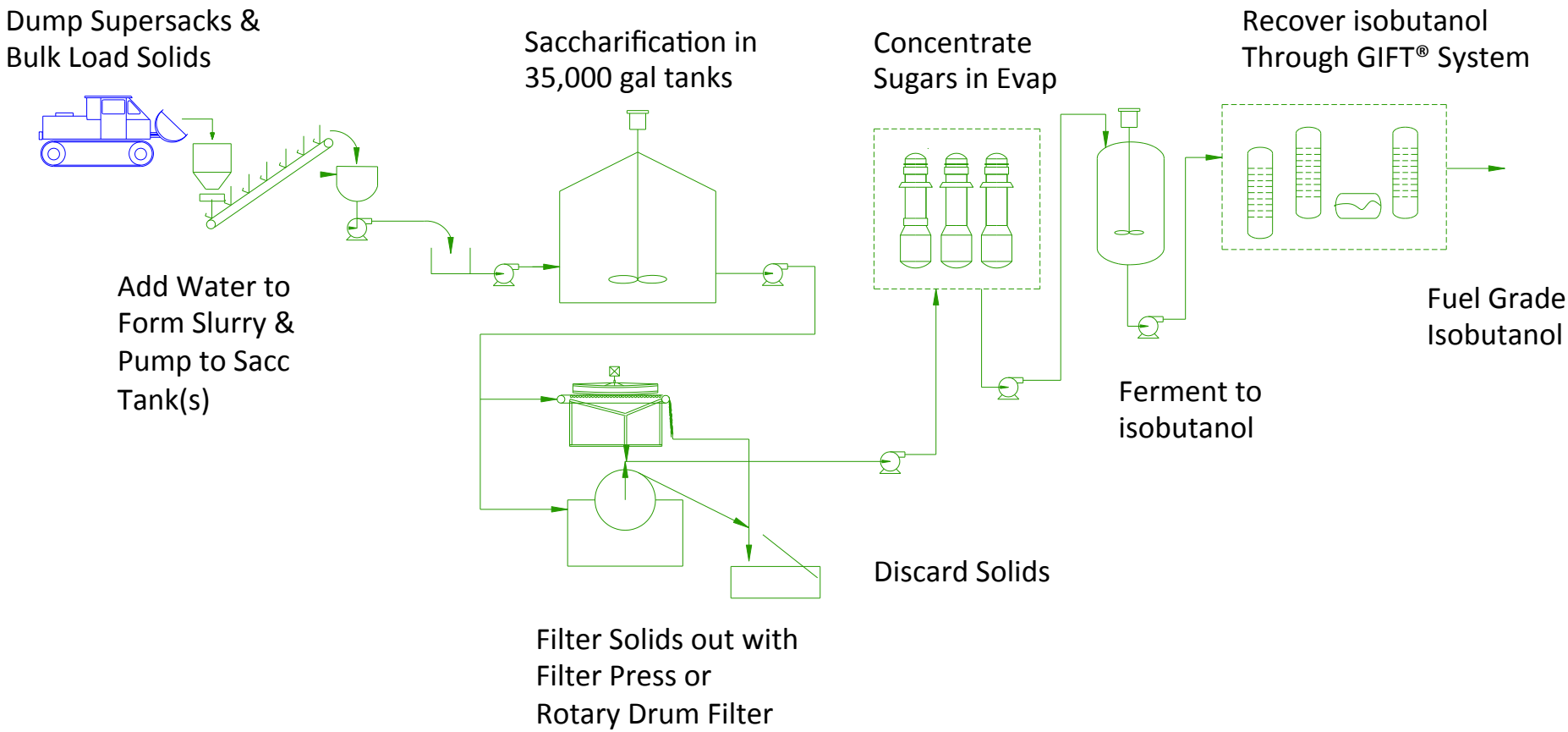
216,000 lb wet
52 Dry tons
265 Supersacks



Northwest Advanced Renewables Alliance

- ICM – St. Joseph, MO
 - Considerable experience with:
 - Biomass Saccharification
 - Isobutanol Fermentation, Recovery and Purification using Gevo Technology
 - Existing, 35,000 gallon enzyme saccharification tanks
 - Filter Press with washing capability to remove solids after Saccharification
 - Evaporation to concentrate sugars to 150 g/l for fermentation
 - 3 Existing 6,000 gallon sterile fermenters that can be used in parallel
 - Existing pilot unit using the Gevo GIFT technology for high efficiency of isobutanol recovery from Fermentation

Process Flow for Enzyme Sacc & Fermentation at ICM



Loading the Solids

NARA – USDA Update, Washington, DC
November 17, 2016



Supersacks being Dumped
and Scooped



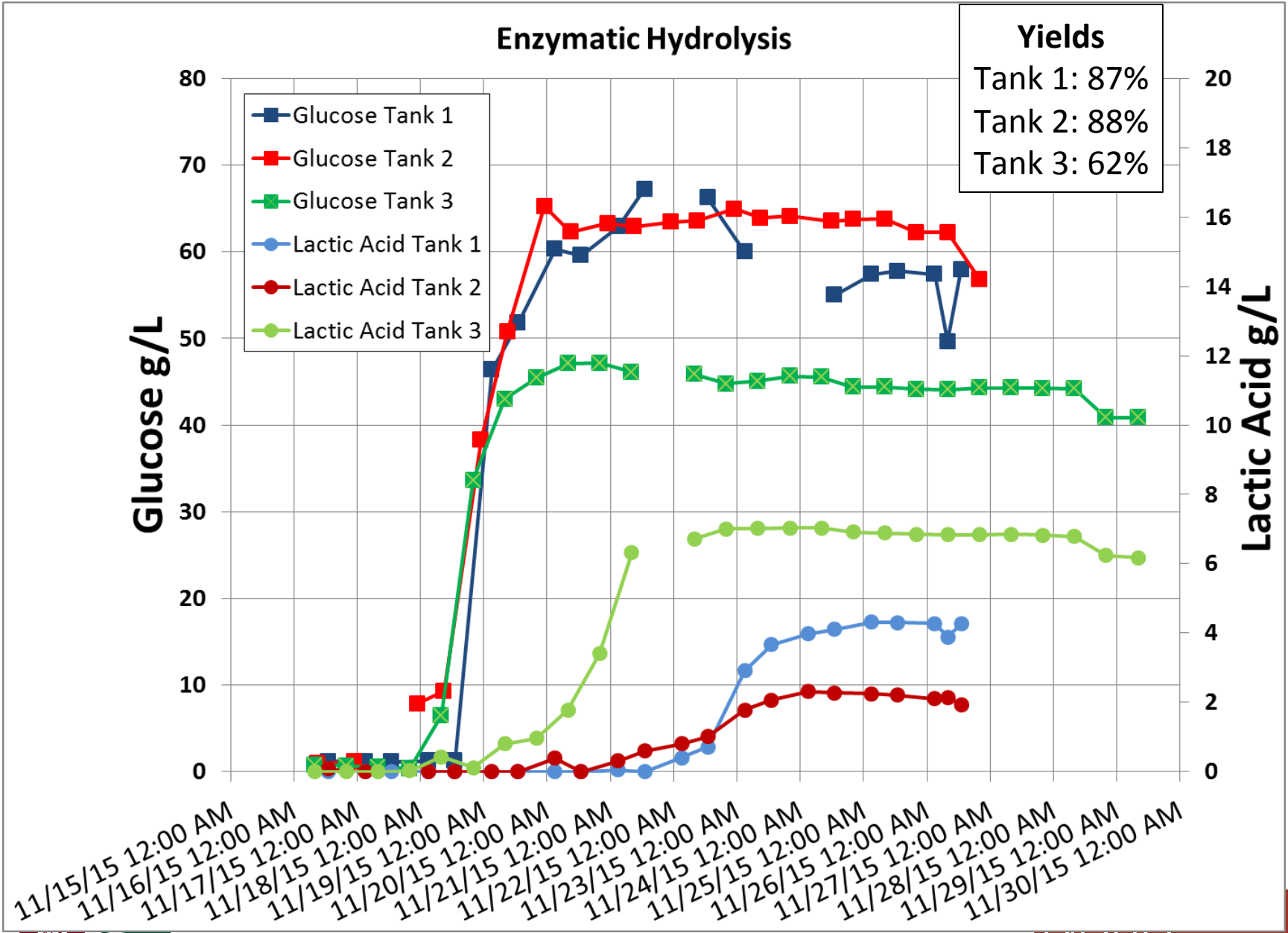
Slurry That
Was Pumped



Loading
Cosmo



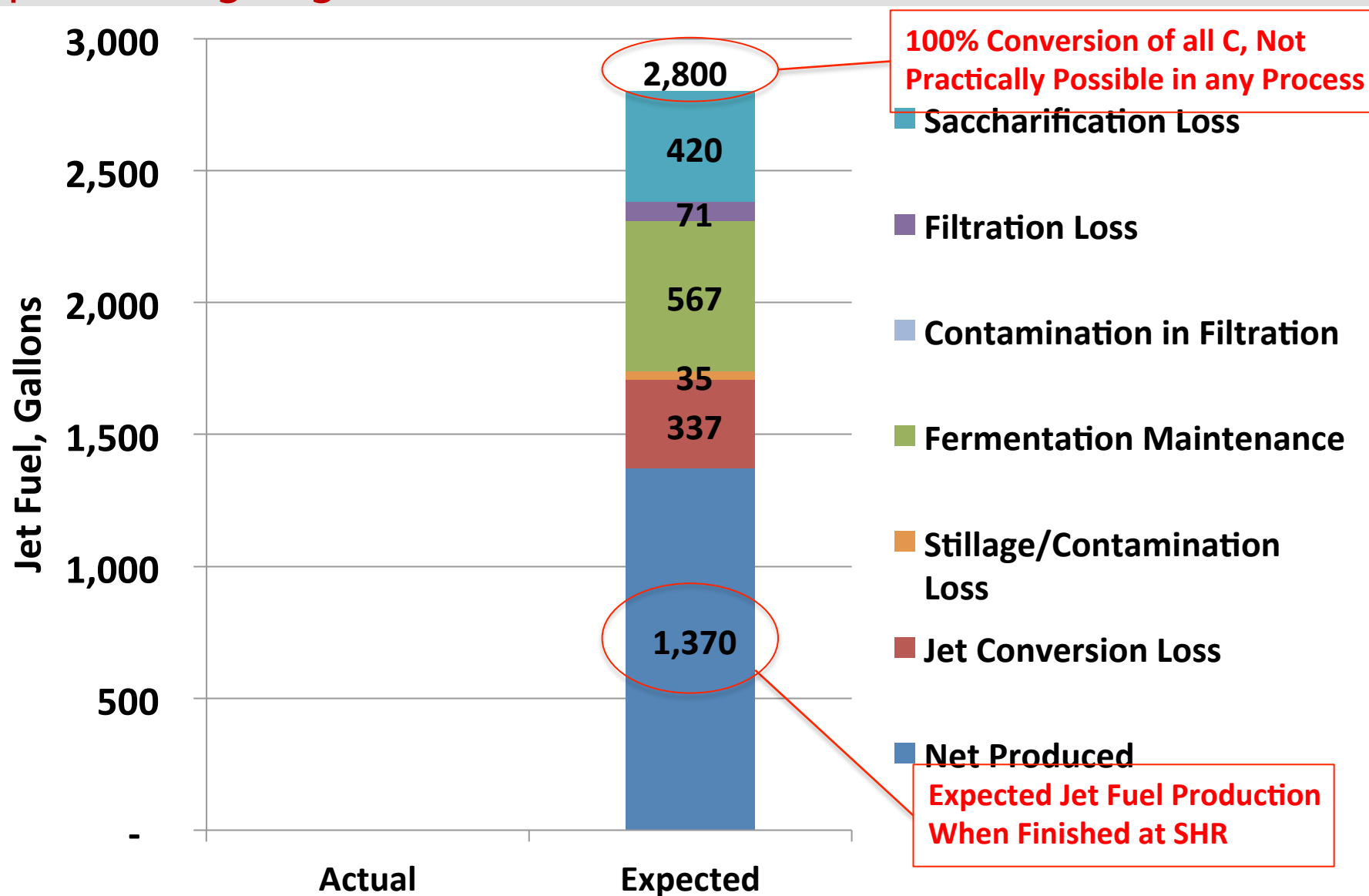
Enzymatic Hydrolysis Excellent, but ...



- Filtration of Post Saccharification Solids
 - Testing immediately before run indicated there might be issues
 - Filter Press Filter Rate was much slower than expected
 - Rotary Drum Filter was much slower than expected
 - Sugar losses were much greater than 10% expected
- Led to Long Storage of Sugar
 - Stored at high temperature to avoid contamination
- GIFT system could not maintain design pressure
- Distillation upsets led to contamination of product

Expectation going into ICM as Potential for Jet Fuel

NARA – USDA Update, Washington, DC
November 17, 2016

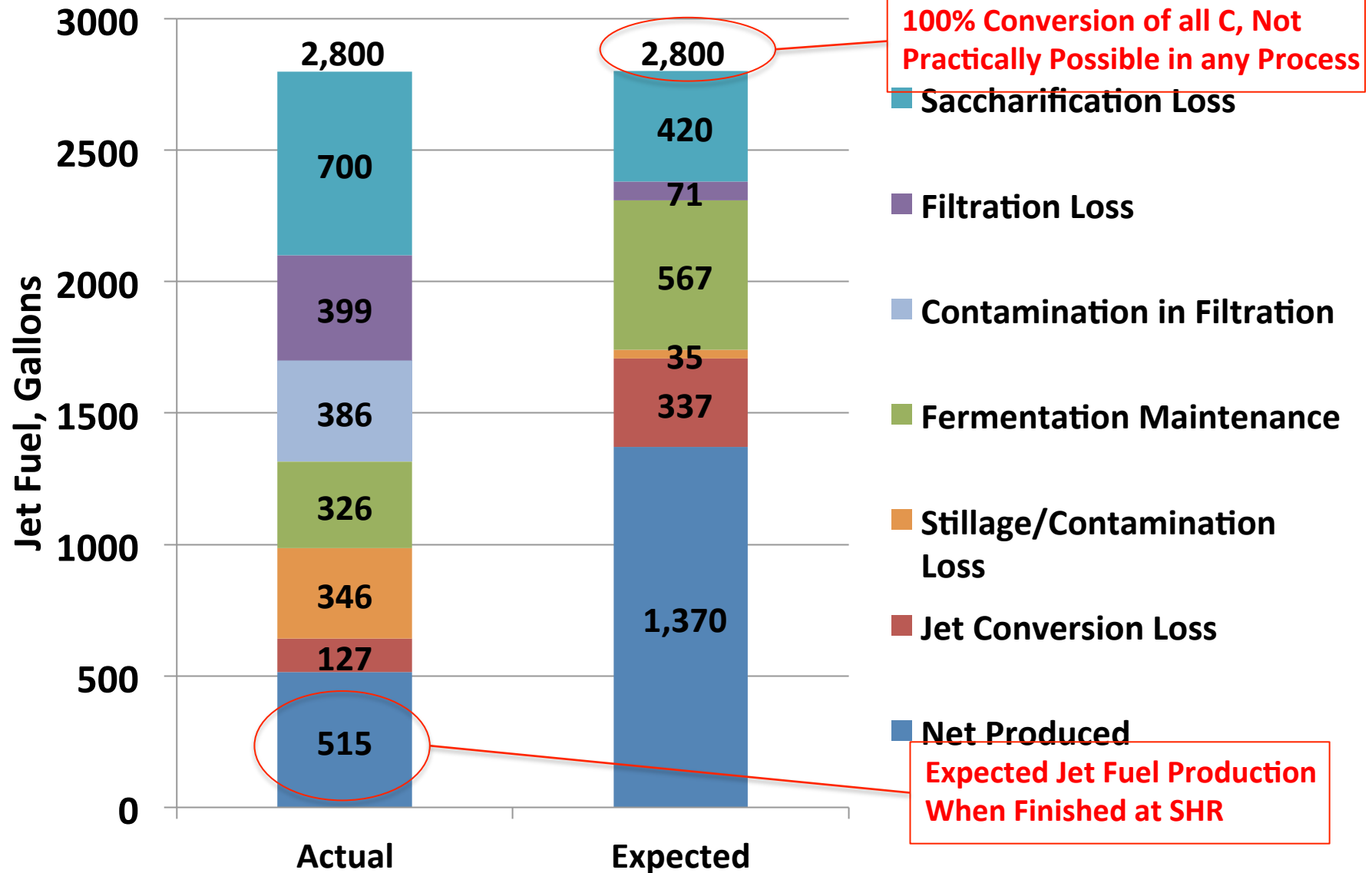


Losses in Ferm & Jet Conv are based on % of material, so design is based on larger amounts coming into those areas



Northwest Advanced Renewables Alliance

Expectation going into ICM and Result as Potential for Jet Fuel



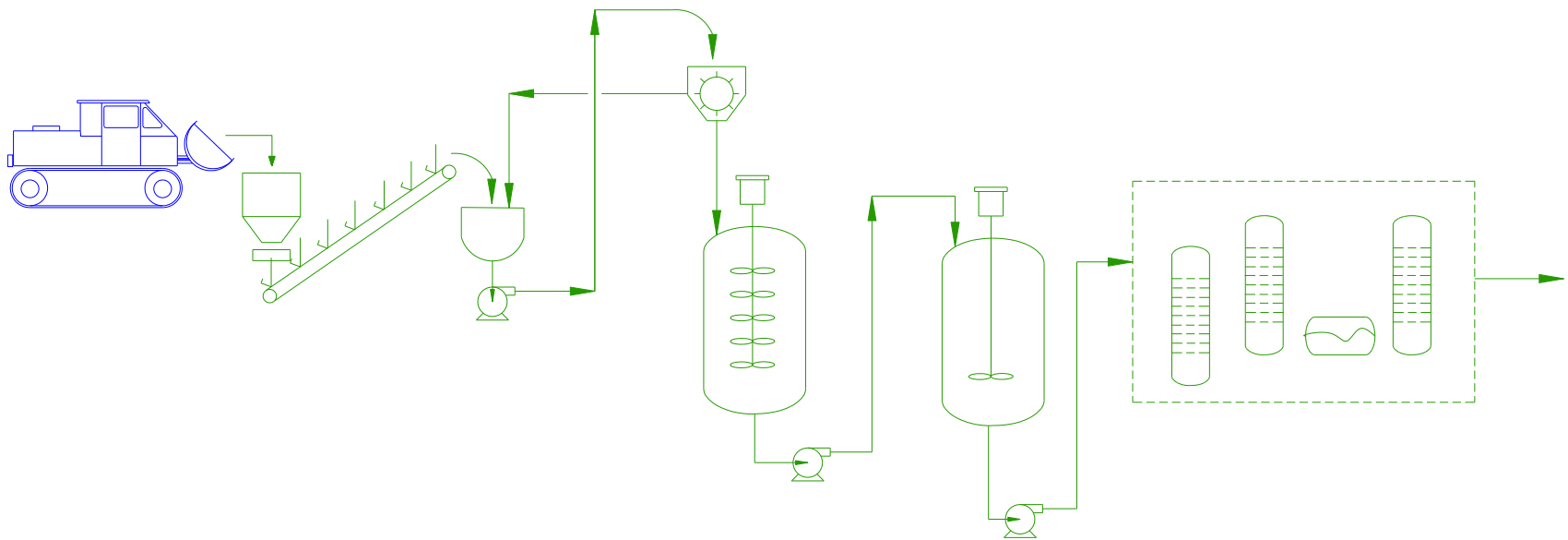
Losses in Ferm & Jet Conv are based on % of material, so design is based on larger amounts coming into those areas



Northwest Advanced Renewables Alliance

- Pretreated Feedstock Exhausted
- Additional Cosmo Specialty Fibers Material Reject Pulp Might be Available
- Rejected Pulp from Cosmo Specialty Fibers
 - Cosmo material was part of the ICM run
 - ~ 10,000 lb was evaluated in 1st Phase
 - Process is Bisulfite & Similar to NARA/SPORL
 - Feedstock is Hemlock from the NorthWest
 - Retrofitting a Mill or utilizing residues is a design consideration of NARA

- Process Material through Fermentation without Filtering
 - More like a NARA envisioned Commercial Process
 - Requires more Dilute Fermentations
 - Solids could be an issue in fermenters and GIFT



Results with Cosmo Reject Pulp

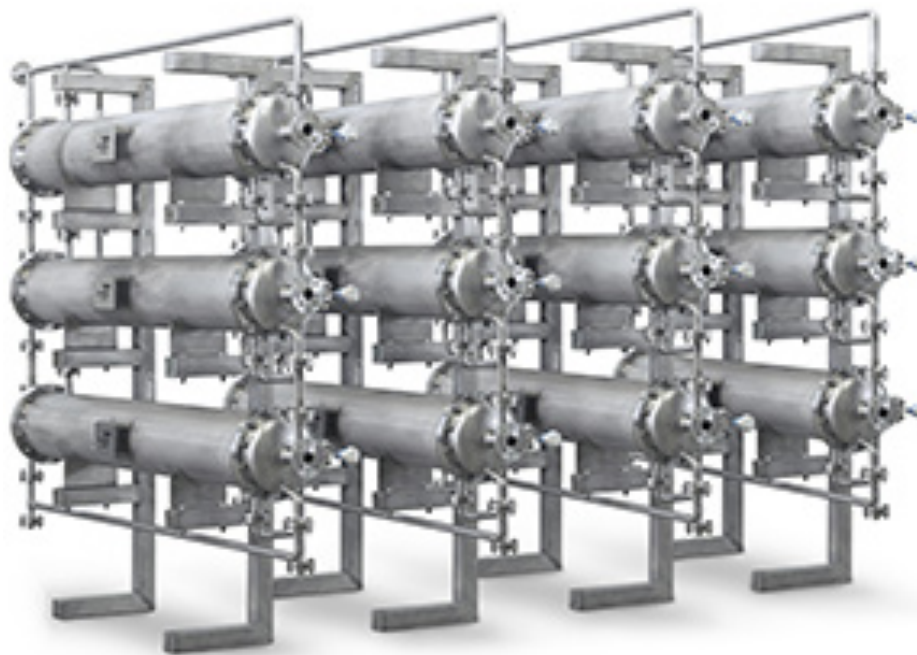
NARA – USDA Update, Washington, DC
November 17, 2016

- After a rough start & initial contaminated saccharification
- 4 Successful Fermentations
- Volume of iBuOH sufficient to produce 1,000 gallons of Biojet.
- Final removal of water before producing Jet



Final Water Removal

- Distillation System at ICM was insufficient to remove Final Water
- Membrane Option was available from Whitefox Technologies
- iBuOH was shipped to Calgary, Alberta for final processing.



Lessons Learned

- Trial as many operations in Toller Equipment as Possible
- Don't Push ahead with Unit Operations Until you are sure the next Unit will Perform as Expected
 - Especially if there could be issues with storing the intermediate
 - This Occurred when we had all solids enzymatically hydrolyzed or in process before we knew how the solids separation would perform
- Avoid storing intermediates which are subject to degradation
 - Moderate concentration sugar streams which are subject to contamination under some conditions and degradation under other conditions
- Make sure there is a strong cost control plan at the toller
 - Fixed Price Might be Best
 - Fixed Rates for tons fed possibly

- South Hampton Resources, Silsbee, TX
 - Biojet Production
 - Only pilot facility already set-up to produce BioJet from isobutanol
 - Facility has been running nearly continuously producing Biojet from isobutanol since late 2011 for Gevo

Gevo's Biojet Facility at South Hampton Resources

NARA – USDA Update, Washington, DC
November 17, 2016

South Hampton Resources
An Independent, Petrochemical
Manufacturer in Southeast Texas



Biojet Demonstration Plant
South Hampton Resources
Silsbee, Texas



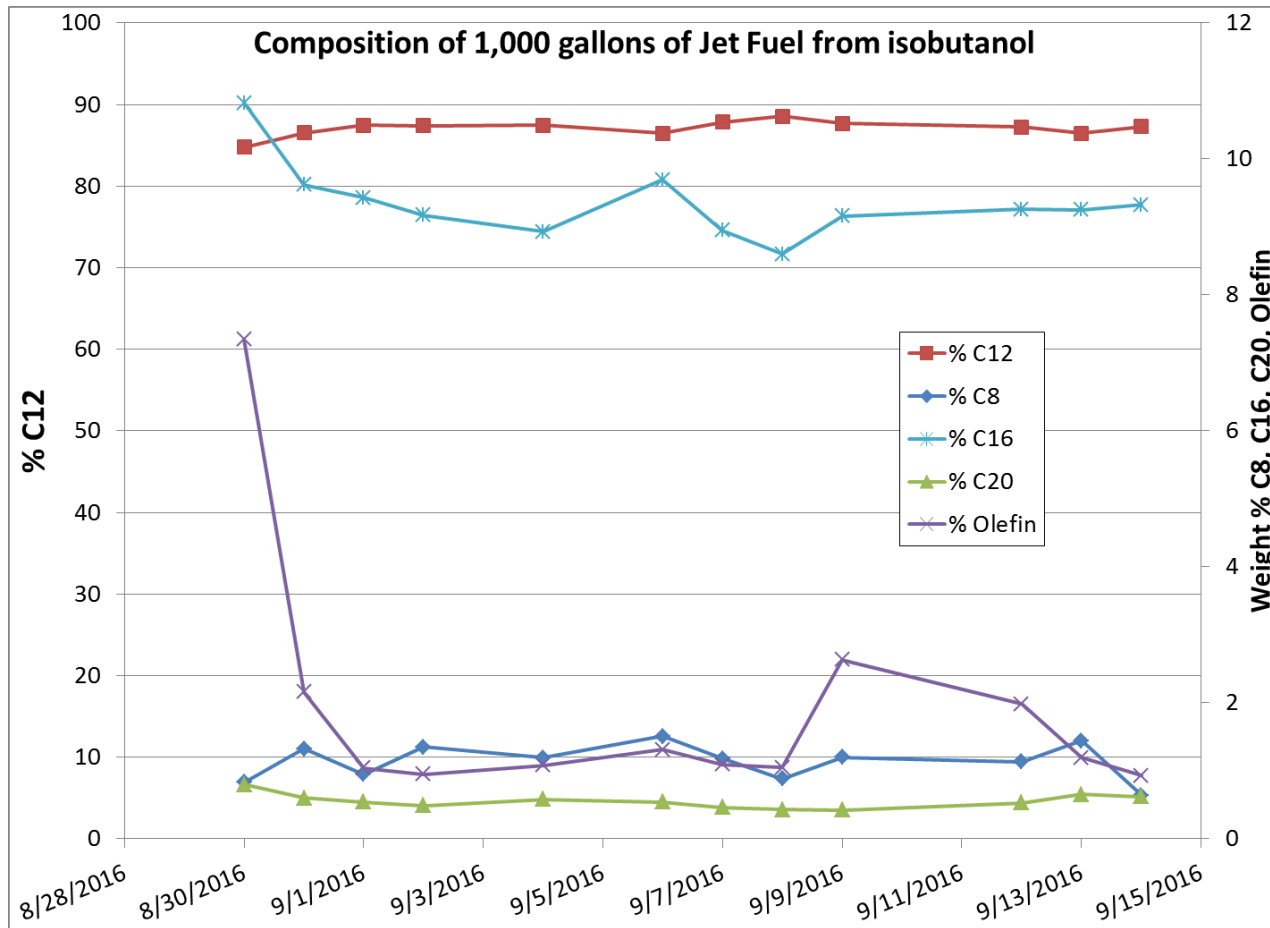
NARA

Northwest Advanced Renewables Alliance

Flawless Operation at South Hampton

NARA – USDA Update, Washington, DC
November 17, 2016

- Conversion from isobutanol took about 15 days to complete



IAC Port Arthur
6175 Highway 347
Beaumont, Texas 77705-7657 United States of America
T: 409-212 9322
F: 409-212-9327



Certificate of Analysis

Vessel / Shore Tank :	Submitted Sample	Sample Submitted By :	South Hampton Refining --
Product :	BioJet	Analysis Performed By :	IAC Port Arthur
Client Reference :		Date Sampled :	15-Sep-2016
Terminal / Port / Office:	South Hampton Refining -- Silsbee, TX	Date Reported :	04-Oct-2016
Job ID :	577508-16-0041472	Submission ID :	008-1603881
Comments :	Serial# 244585, 244614 ,& 244601 (Lot# F02SF40001)		

Method	Test	Submitted		
		Sample Number	Result	Pass-Fail
ASTM D3242	Acid Number , mg KOH/g	008-1603881-01-006	0.000	Passed
ASTM D86	Observed Barometric Pressure , mm Hg / kPa		760 / 101.3	
	Initial Boiling Point , °C		163.2	
	5% Recovered , °C		175.8	
	10% Recovered , °C		176.4	
	20% Recovered , °C		177.3	Passed

Final 1,000 Gallons of Fuel Passed the Specification Established in ASTM D7566

**ASTM D7566 was adopted by the Aviation Industry through
great leadership efforts of NARA partner - Gevo**





Blending the fuel

Fueling the Alaskan Airline 737 at
Seattle Airport
Monday Morning November 14th



The Project was Funded by the U.S. Department of Agriculture's National Institute for Food and Agriculture Program, Award No. 2011-68005-30416.

Novozymes Generously Contributed the Cellic® CTec3 Used for the Enzymatic Saccharification

Hard work and dedication of the Engineers and Technicians at Lane, Zeachem, ICM, Whitefox, SHR and the NARA 1,000 Gallon Task Team

Commercial Demonstration

NARA – USDA Update, Washington, DC
November 17, 2016



Gevo has made possible these flights by leading the effort to obtain the ASTM Certification of this fuel