

# **The Application of NIR in Ethanol Industry**

## ----- A Dry Lab for Ethanol Plants

National Corn-to-Ethanol Research Center  
Analytical Laboratory  
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# READY for RESEARCH

The National Corn-to-Ethanol Research Center,  
Edwardsville, IL has opened its doors to those  
who want to test the latest ethanol technology.



# PILOT PLANT: Dry Grind and Wet Mill



- Is flexible and capable of operating with a variety of processes and feedstocks.
- Performs near-term industrial research.



- Training center for current and future personnel in biofuels industry.
- Produces robust and accurate data



# Analytical Lab

- Quality Control
- Commercial Testing
- R & D



# Fermentation Lab

- Small-scale screening of products and ideas.
- Screening and optimization studies using experimental design software
- Ethanol yield validation

# Near Infrared Spectroscopy (Basic)

- **Near Infrared Spectroscopy is used where multicomponent molecular vibrational analysis is required in the presence of interfering substances. NIR becomes a valuable tool for qualitative and quantitative analysis with the development of chemometric methods and fiber optics in the 1980s.**
- **Application: food industry, clinical investigations, environmental monitoring, biotechnology etc.**
- **Quantitative analysis: the instrument response needs to be calibrated with reference data obtained by conventional chemistry methods.**
- **Samples format: solid, liquid (water), and between.**



# Near Infrared Spectroscopy (Application in Ethanol Industry)

## Advantage

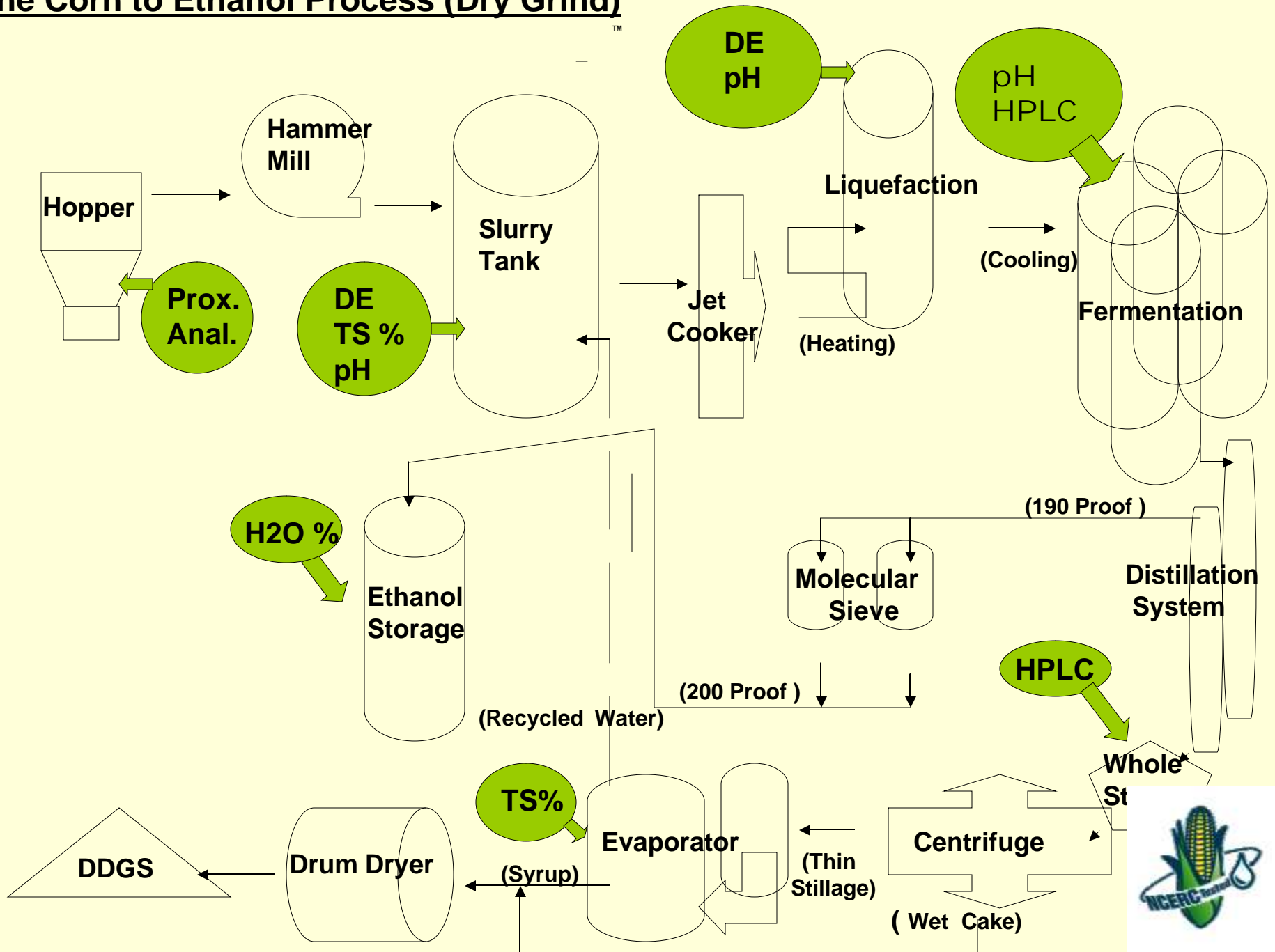
- Save manpower; Save time; Save cost
- Enhance technical capability
- Reduce data variability

## Disadvantage




- Wet chemistry method standardization
- Sample coverage
- Instrument stability  
etc.



# The Corn to Ethanol Process (Dry Grind)

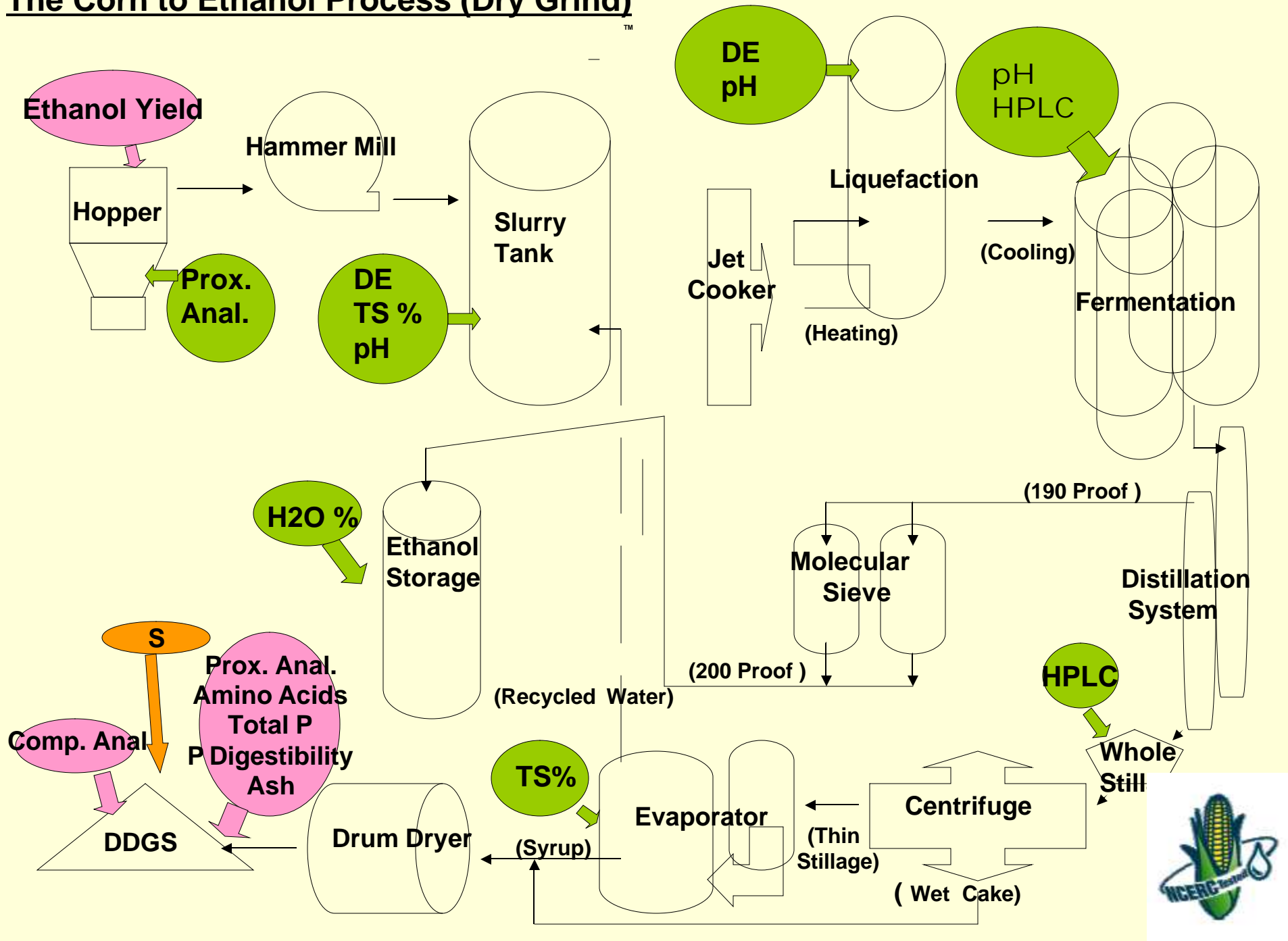


# Quality Control Screening

Process	Tests	Sample Specification	Green 	Yellow 	Red 
Liquefaction	DE	Before Jet Cooker	5 – 10	3 – 5 or 10 - 15	> 15
	TS %		28 – 33	25 - 28 or 33 – 36	> 37 or < 25
Fermentation	pH		3.5 – 4.3	3.2 – 3.5 or 4.3 – 4.5	> 4.5 or < 3.2
	HPLC (lactic acid)	After 30 hours of fermentation	< 0.2	0.2 – 0.25	> 0.25
Final Ethanol	KF (H <sub>2</sub> O %)		< 1.0	1.0 – 1.5	> 1.5
DDGS	Moisture (H <sub>2</sub> O %)		8.0 – 13.0	7.0 – 8.0 or 13.0 – 15.0	> 15 or < 8.0



# The Corn to Ethanol Process (Dry Grind)



# Analytical Laboratory at NCERC

**Acid Detergent Fiber (AOAC 978.18)**

***Acid Detergent Lignin (978.18)\****

**Amino Acid Profile (19 amino acids)**

**Crude Fat (AOAC 945.16, AFIA recommended)**

**Crude Fiber (AOAC 978.10, AFIA recommended)**

**Crude Protein (AOAC 990.03, AFIA recommended)**

***Lysine Digestibility\****

**Moisture (NFTA 2.2.2.5.)**

***Mycotoxins (30 or so toxins)\****

**Neutral Detergent Fiber (AOAC2002.04)**

***Phosphorus Digestibility\****

**Phytate (AOAC 986.11)**

**Starch (modified AOAC 996.11)**

***Sulfur\****

**Total Phosphorus (AOAC 970.39)**

**\* Method in development**



## Fermentation Laboratory at NCERC

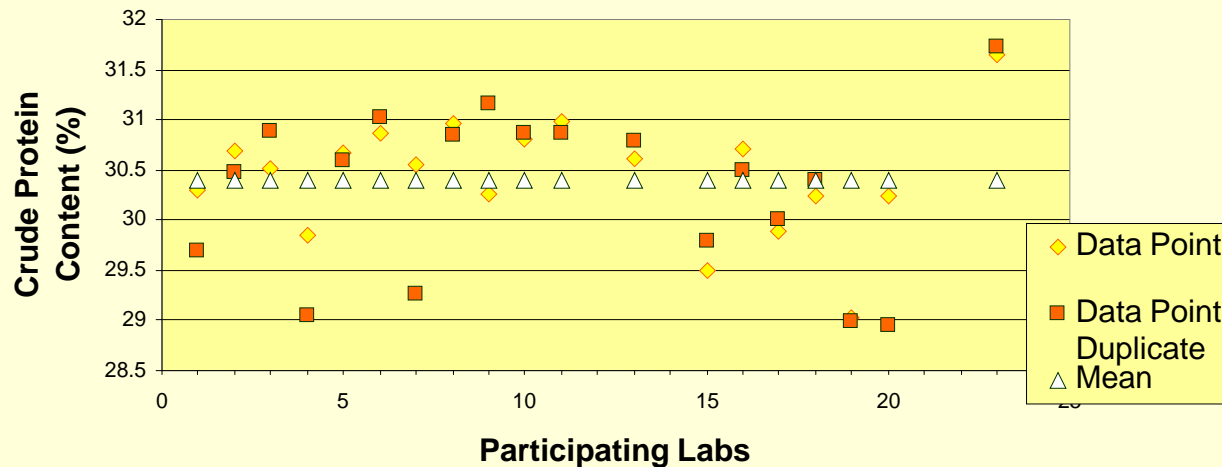
- The fermentation laboratory at the NCERC is developing an NIT calibration for ethanol yield.
- The reference method is the flask fermentation method used at the NCERC for client-based studies.
- Environmentally and genetically diverse samples will be used in developing the calibration.
- Calibration building will begin summer of 2007 with the majority of the testing finished by the end of the year.



# Evaluation of Analytical Methods for Analysis of DDGS

Sponsored by The American Feed Industry Association (AFIA),  
Renewable Fuels Association (RFA), and  
The National Corn Grower's Association (NCGA)

## Data Comparison of NCERC and Other Laboratories (NCERC as # 1; Sample B)



# Sample Coverage

- **New feedstock Products**
  - **New technology**
- etc.**



# Building up DDGS Data Base

## National DDGS Library

Supported by

Illinois Department of Commerce and Economic Opportunity

Illinois Corn Grower's Association

### Table Contributing Ethanol Plants

Plant #	Location	Sample Size (g)
1	MN	1,962
2	IA	11,205
3	ND	5,222
4	IN	2,791
5	WI	2,402
6	IA	5,704
7	IL	4,604
8	MN	3,563
9	IL	1,866
10	MN	4,376
11	IA	1,018
12	IA	1,348
13	IA	280
14	KS	7,834
15	NE	4,306
16	MI	4,589
17	IA	4,732
18	IL	2,096
19	IL	5,240
20	IA	5,847



# DDGS Quality Assurance Program for Illinois Pork Producers

(Submitted to the Illinois Corn Grower's Association)

	Basic Service	Premium Service	Supreme Service
Nutrients Coverage	<p>Crude Protein, Crude Fat, Crude Fiber, Moisture</p>	<p>Crude Protein, Crude Fat, Moisture Neutral Detergent Fiber (NDF)</p> <p>Total Residual Sugars Amino Acids Total Phosphorus Phosphorus Digestibility</p>	<p>Crude Protein, Crude Fat, Moisture Neutral Detergent Fiber (NDF)</p> <p>Total Residual Sugars Amino Acids Total Phosphorus Phosphorus Digestibility</p> <p>Risk factor screen (Mycotoxins, Sulfur)</p>



# The Pilot Plant at NCERC

- **Confidential Client Research**  
**Over 25 client trials conducted**  
**(Custom modifications, custom trials, etc.)**
  -
- **Grant Research in Areas of Unmet Needs**  
**Industry/Research Center Partnerships**  
**Results in Public Domain**



# Future Development

- Application in the plant
- Cellulosic ethanol production
- Biorefinary
- etc.



# Conclusions

1. A non-destructive and rapid NIR instrument can serve ethanol plants as a quality control unit.
2. The robustness of the NIR method relies on the quality of calibration software which requires high accuracy of reference chemistry methods and comprehensive data base.
3. Routine verification by wet chemistry methods is recommended for any NIR instrument.



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