NREL/PO-510-31791
March 2002

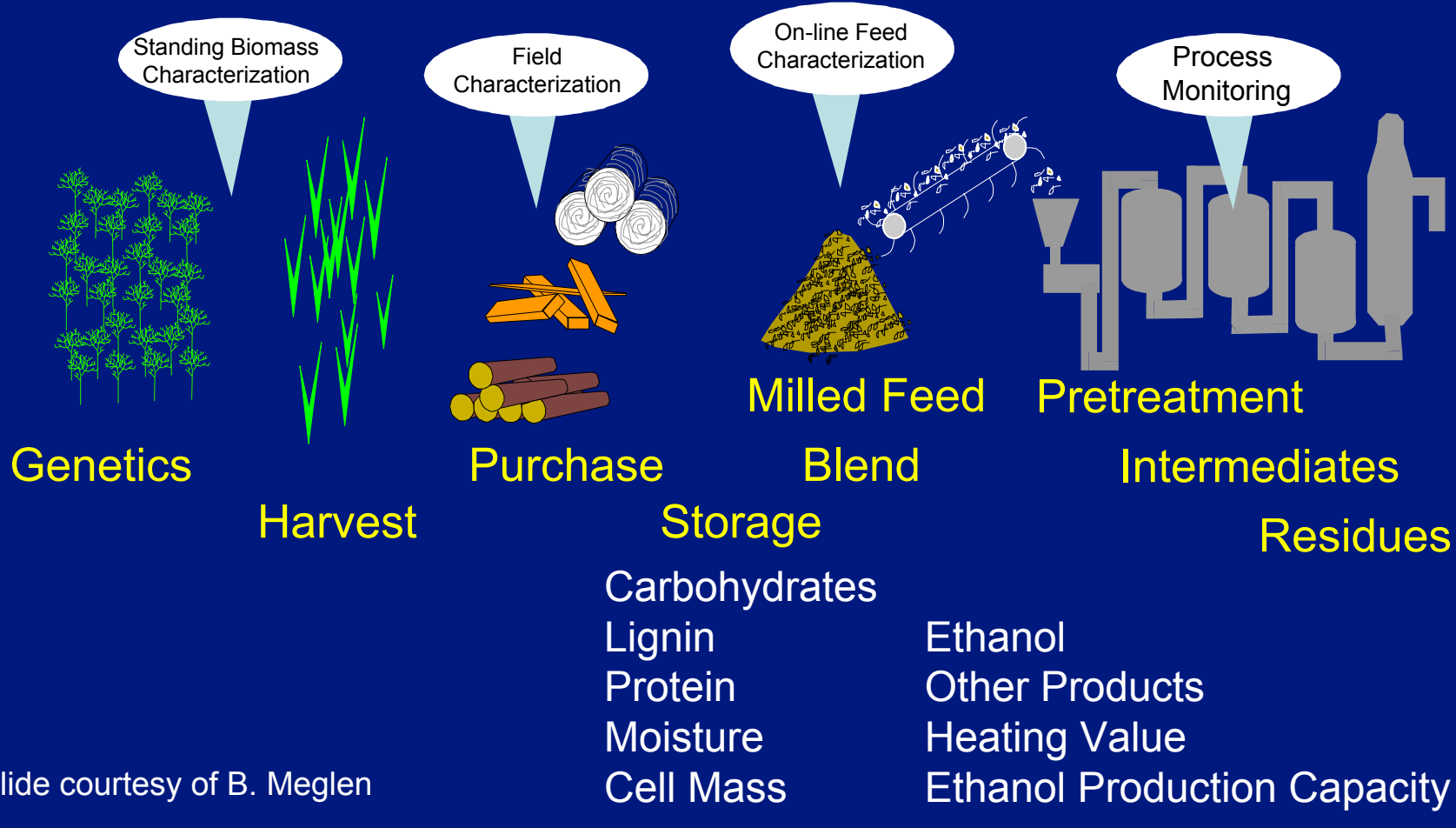
Biomass Analysis at NREL

- Biomass Analysis Experts: more than 20 years of experience
- Experienced in the analysis of a wide variety of biomass types
- Standard Methods published through ASTM E48 Biotechnology Standard Reference Materials available through NIST

Biomass Heterogeneity

- Inherent property of biomass and biomass-derived materials
- Difficult to control
- Possible to monitor
- Accurate analysis requires multiple samples
- Current wet chemical methods are too expensive and slow to be useful in industrial applications
- Rapid and inexpensive compositional analysis methods are important enabling technologies for processes designed to convert biomass to fuels and chemicals

Industrial Application of Rapid Analysis

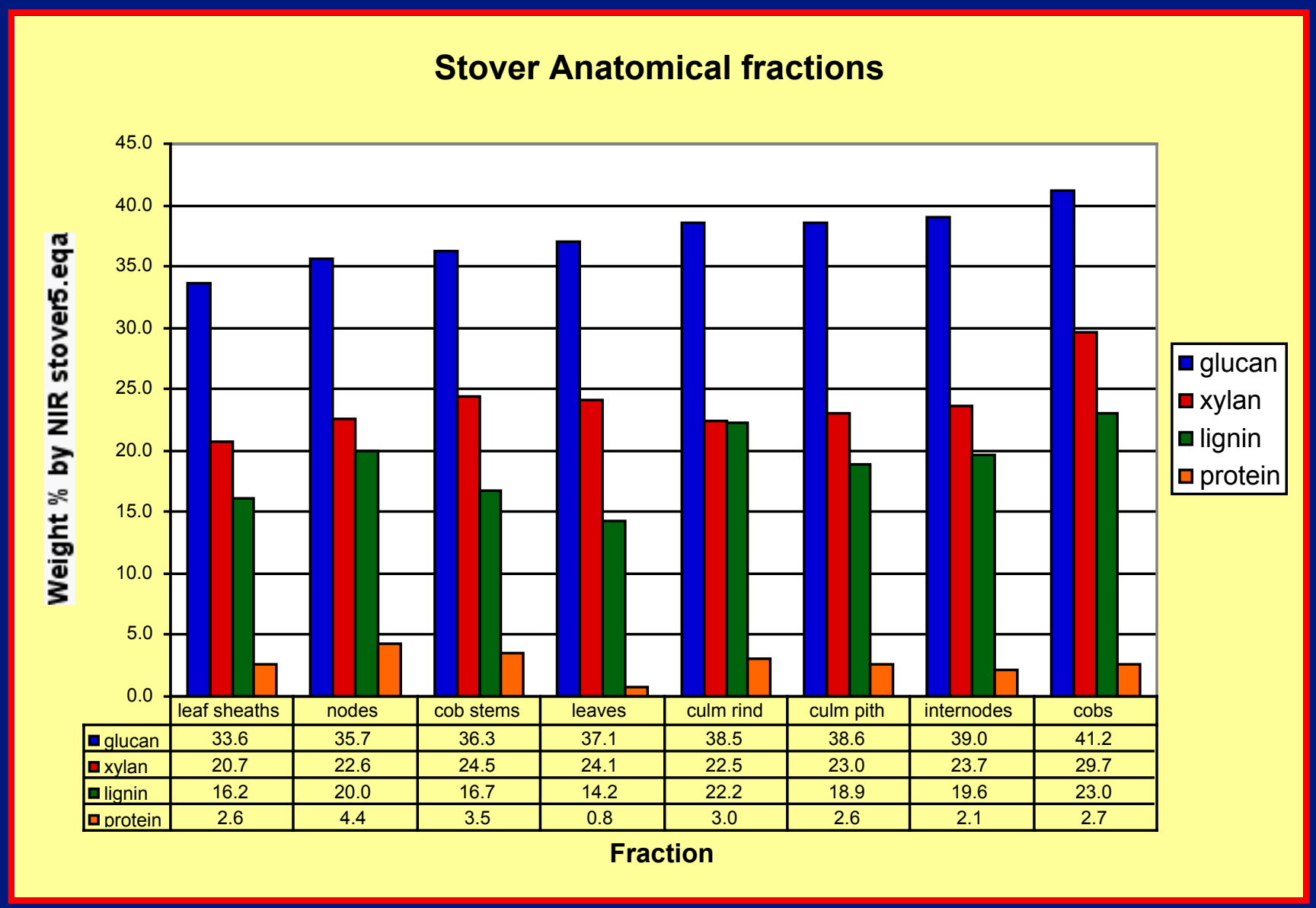


Slide courtesy of B. Meglen

Rapid Biomass Analysis Methods are being developed to support all phases of the biomass conversion process

- Genetics
  - Thousands of plants can be screened for interesting compositional mutations
- Harvest
  - Field monitoring of crops can determine harvest readiness
- Purchase
  - Feedstock price can be based on value instead of weight
- Storage
  - Compositional changes can be monitored as a function of storage time and conditions
- Blending
  - Provides process with more uniform feedstock
- Feedstock composition
  - Allow process to adjust to incoming feed
- Pretreatment
  - Allows reaction conditions to be optimized to incoming feed
- Process monitoring
  - Real-time information for enzyme, organism and nutrient loading
  - Allows reaction conditions to be optimized
- Products
  - Yields and product quality data available in real-time

Chemical Data for Rapid Analysis Calibration

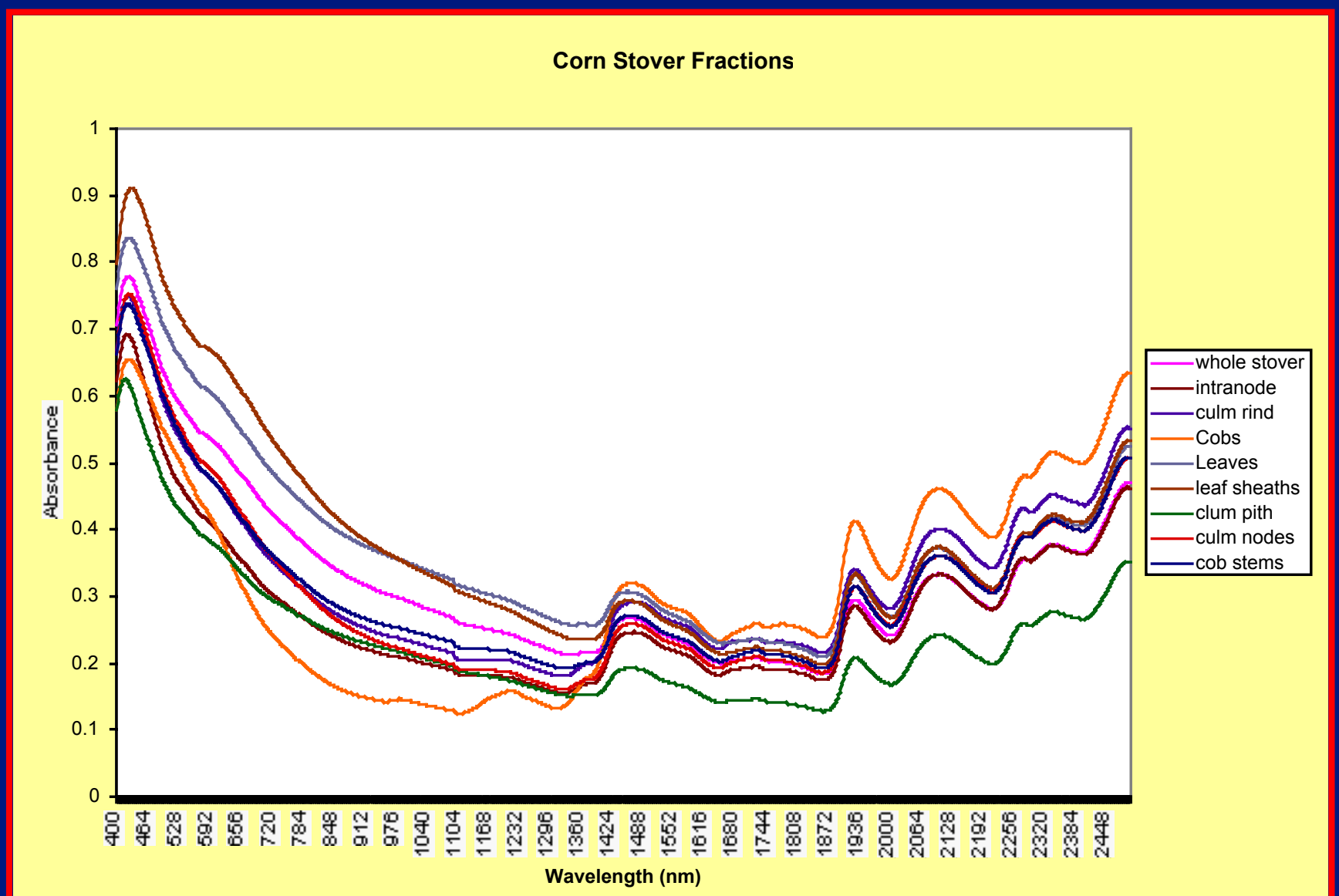


	leaf sheaths	nodes	cob stems	leaves	culm rind	culm pith	internodes	cobs
glucan	33.6	35.7	36.3	37.1	38.5	38.6	39.0	41.2
xylan	20.7	22.6	24.5	24.1	22.5	23.0	23.7	29.7
lignin	16.2	20.0	16.7	14.2	22.2	18.9	19.6	23.0
protein	2.6	4.4	3.5	0.8	3.0	2.6	2.1	2.7

Rapid Analysis Calibration Samples

- Should resemble samples to be analyzed
  - Similar compositional ranges
  - Independent compositional variance
- Should take into account multiple sources of compositional variance
  - Regional effects
  - Seasonal effects
  - Harvesting times and methods
  - Storage
  - Degradation
  - Genetics

Spectroscopic Data for Rapid Analysis Calibration

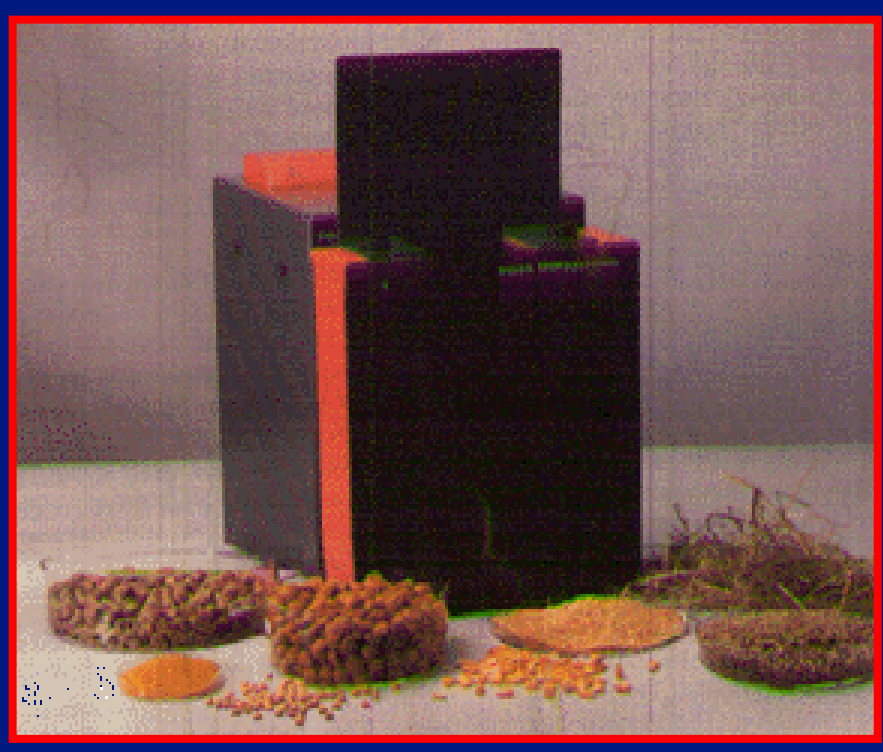


Spectroscopic Data

- Rapid technique that contains compositional information
- Quality, reproducible spectra are essential for robust method
- Spectral collection methods must be robust in field and/or industrial settings
- Speed and cost of rapid analysis method determined by spectroscopic method and equipment selected

NIR Spectrometer

- Bench-top system
- Designed for forage and grain analysis
- Accessories for natural products
- Calibrations easily transferable to other instruments



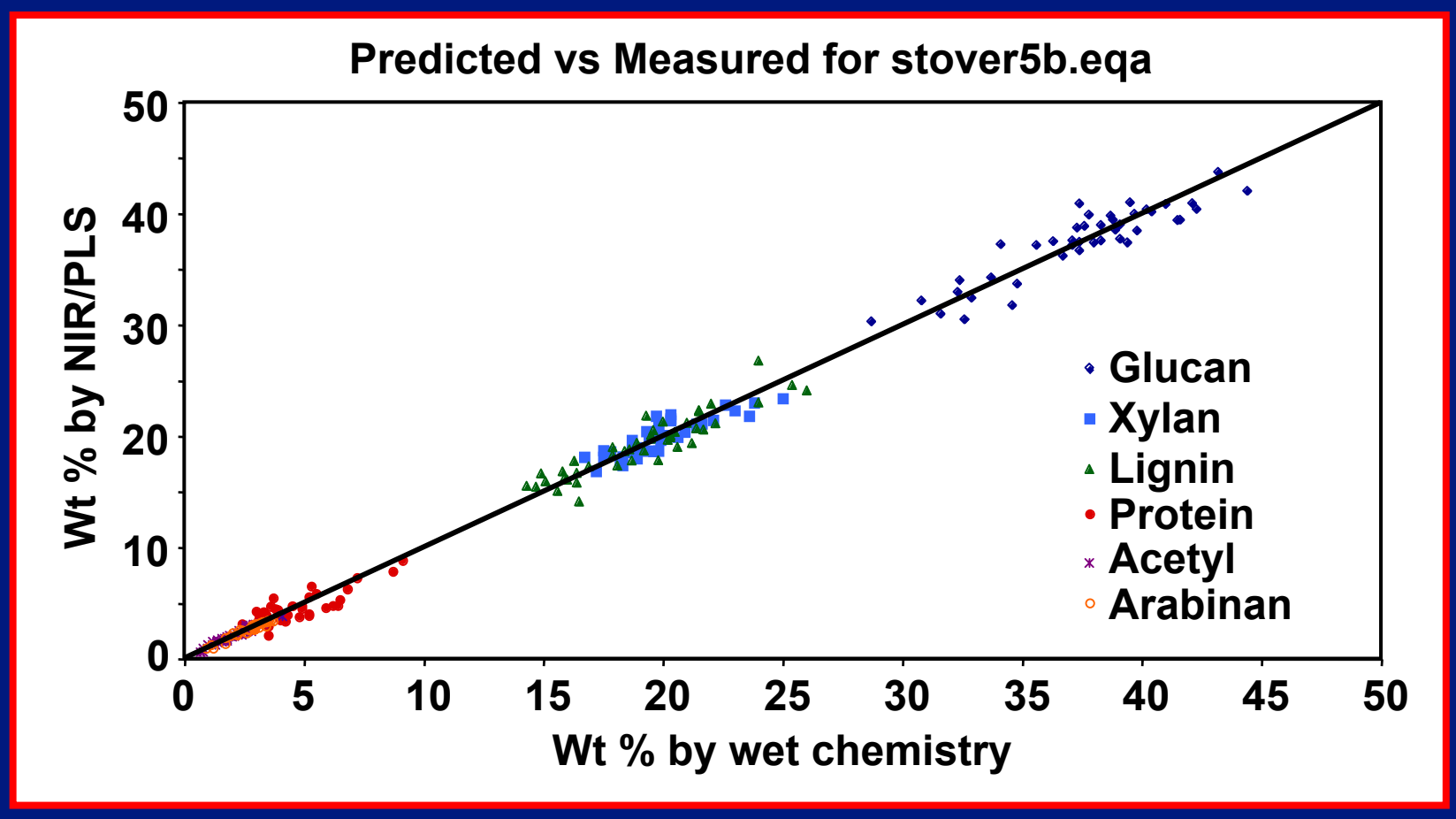
Foss NIR6500 Forage Analyzer

http://www.foss-nirsystems.com

Field Mobile NIR Spectrometers

http://www.asdi.com/

NIR/PLS Compositional Analysis
Corn Stover Feedstocks



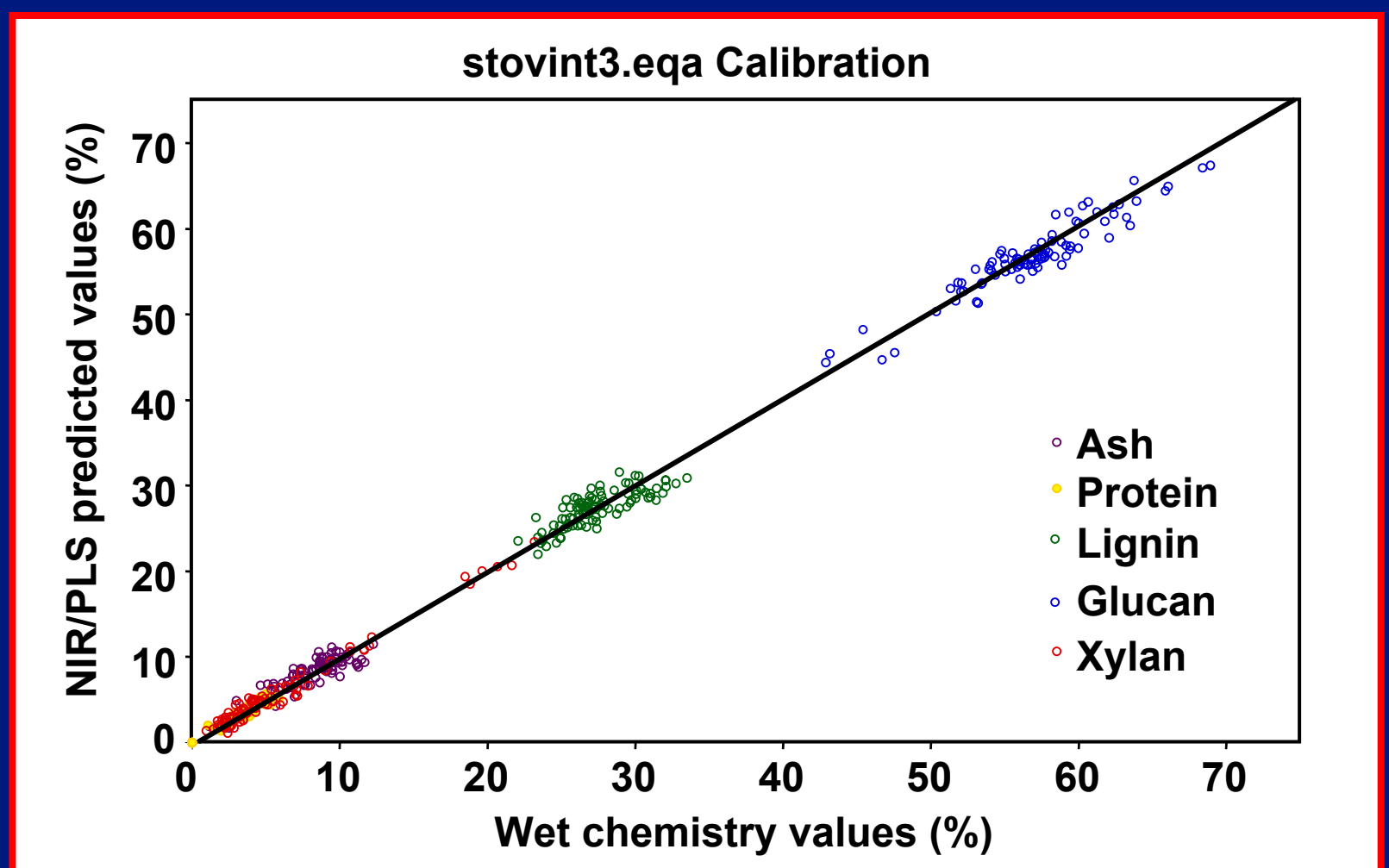
Equation Statistics stover5b.eqa

Constituent	#of PCs	SECV	1-VR	Calibration Range
Glucan	5	1.4	.832	26.9 - 47.9
Xylan	7	1.5	.734	14.5 - 25.3
Arabinan	6	1.5	.909	0.8 - 4.7
Protein	5	0.8	.740	0.1 - 9.19
Ash	7	1.0	.836	0.1 - 13.6
Acetyl	7	0.2	.932	0.1 - 4.8
Lignin	5	1.1	.844	10.8 - 27.4
Mannan	7	1.5	.820	0.1 - 1.5
Uronic acid	7	1.5	.754	2.2 - 4.4

47 samples

Average mass closure 98.3 ± 2.8

NIR/PLS Compositional Analysis Stover Process Intermediates



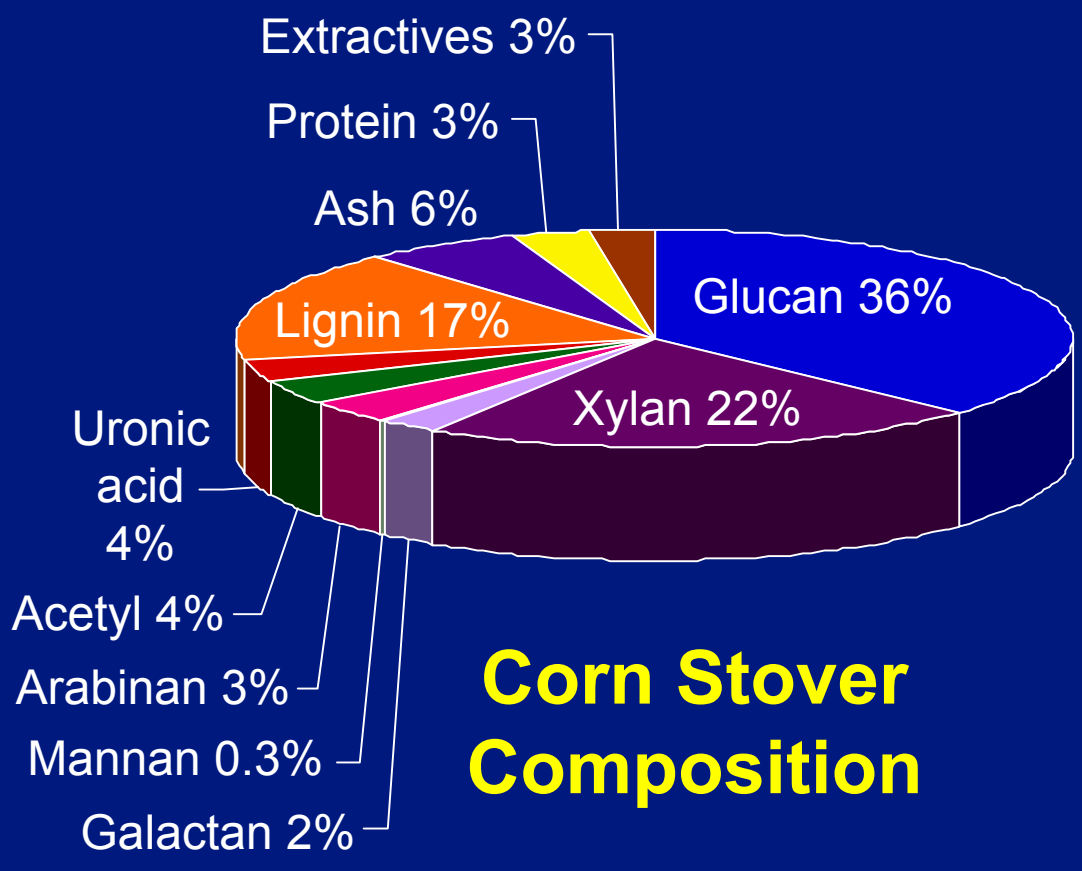
Equation Statistics stovint3.eqa

Constituent	#of PCs	SECV	1-VR	Calibration Range
Glucan	3	1.7	.872	39.6 - 62.1
Xylan	3	1.1	.968	2.8 - 23.1
Lignin	5	1.1	.875	20.2 - 30.0
Ash	3	1.5	.569	0.1 - 11.5
Protein	3	0.5	.782	3.2 - 5.9

96 samples

Average mass closure 101.5 ± 2.8

Corn Stover Feedstock Assessment Average Composition



Corn Stover Composition

- 2000 Harvest data shown
  - 300 samples
  - Four locations
- 2001 harvest study in progress
  - 1000 samples
  - Samples from across U.S corn belt

Improving Research More Samples — More Data

- Corn stover feedstock assessment
- Requires analysis of thousands of samples
  - Not possible using traditional methods
    - Too expensive by wet chemical methods
      - $2,000/ sample x 1,000 samples = $2,000,000
  - NIR/PLS
    - $20/sample x 1,000 samples = $20,000
  - Too slow by wet chemical methods
    - Years to process 1,000 samples
  - NIR/PLS
    - 2-3 days to process 1,000 samples
- Monitoring changes in composition caused by
  - Genetics
  - Harvest year
  - Location
  - Harvest time
  - Harvest method
  - Storage
- Composition reported as average with range of variance

Summary

- Significant savings in time and money
- No loss of precision or accuracy
- Applicable to a wide variety of biomass and biomass-derived products
- NREL uniquely situated for method development
- Supporting current NREL research efforts
- Supporting industrial partnerships
- Enabling technology for biomass utilization

Acknowledgements

- U.S. Department of Energy
- Office of Fuels Development
  – Biofuels Program
- National Renewable Energy Laboratory
  – National Bioenergy Center

  – Contact: bonnie_hames@nrel.gov

Advantages of NIR/PLS Rapid Biomass Analysis

- Faster
  - Minutes instead of days
  - Minimal sample preparation
- Cheaper
  - About $10 per sample
  - Compared to $800–$1,000 for wet analysis
- Better
  - Calibrated using best methods
  - Less operator dependent
- Useful in industrial applications

Rapid Analysis Essentials

- Calibration Samples
- Chemical Characterization
- Rapid Technique
- Multivariate Analysis
- QA/QC