Grain Quality LABORATORY

NIRS Programs That Give Accurate Data for Selections, Science and Marketing

Iowa Grain Quality Initiative

Soybean Breeders Workshop

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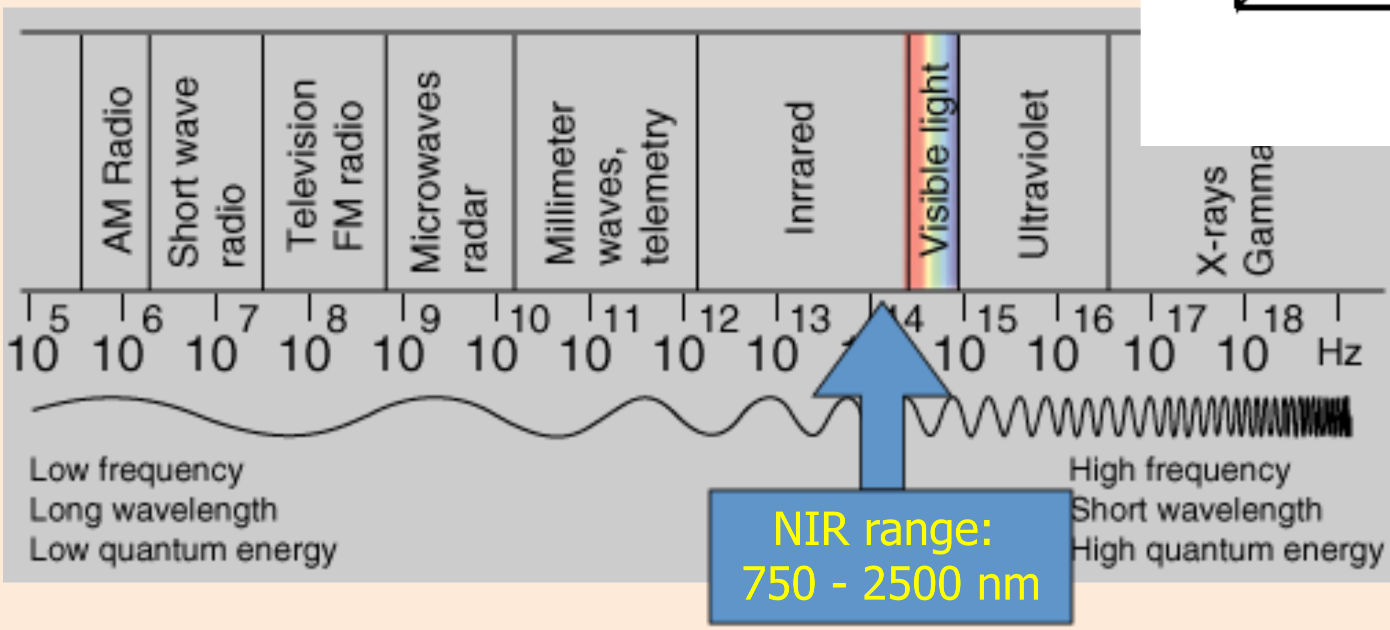
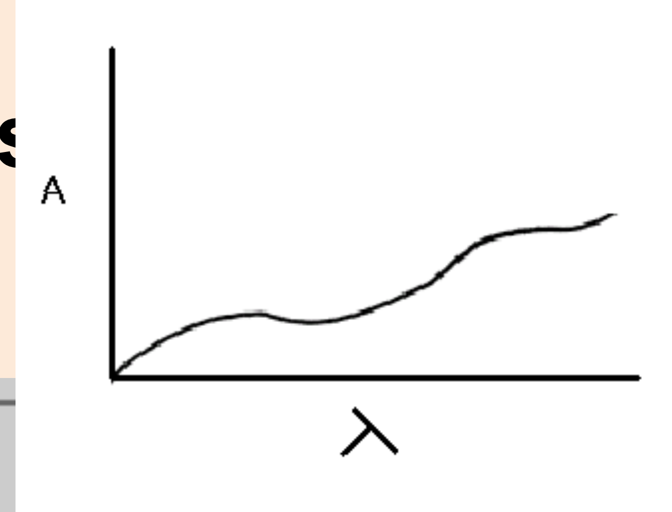
Why NIRS Works

- **Electromagnetic spectrum**

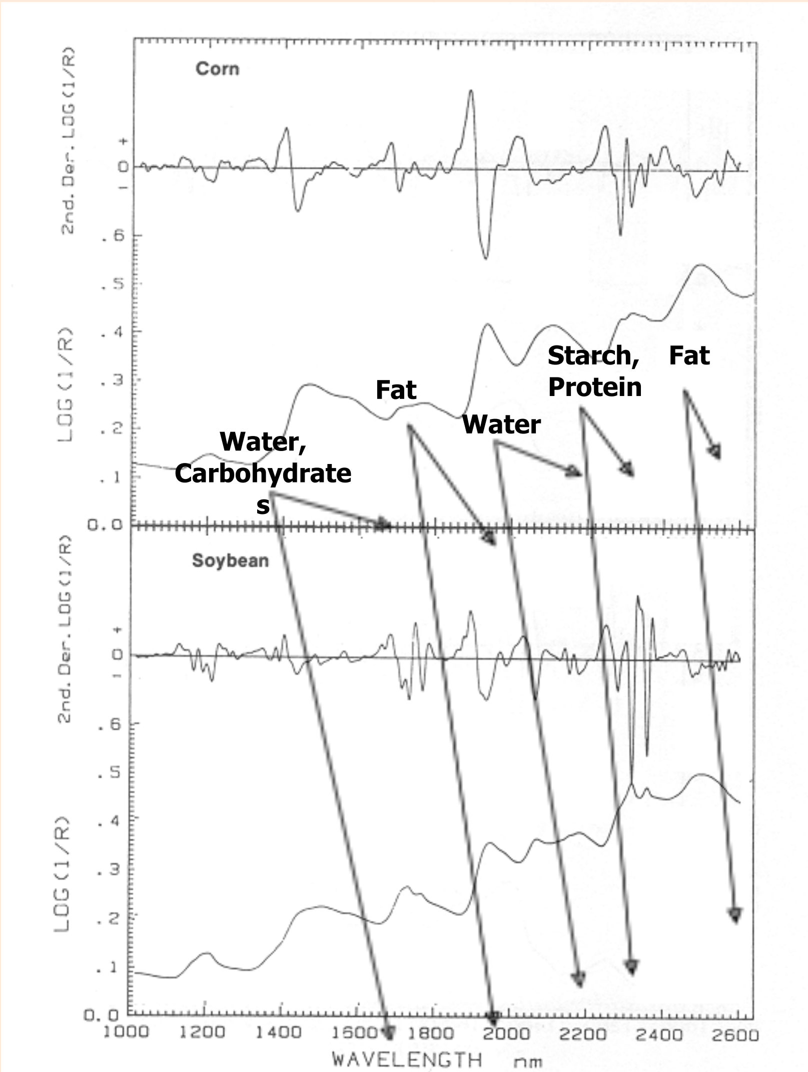




- **What is so special about the waves in NIR range?**

NIR light is absorbed by molecules containing C-H, N-H, and O-H groups (fats, proteins, carbohydrates, organic acids, alcohol, water)

NIRS reflectance spectra and absorption



Important absorptions and their tentative assignment* for food constituents

Wavelength (nm)	Constituent	Assignment
1200	Lipid	C—H
1440	Water and carbohydrates	O—H
1730	Lipid	C—H
1780	Lipid	C—H
1940	Water	O—H
1980	Protein	N—H
2080	Carbohydrates	O—H
2180	Protein	C=O, N—H
2320	Lipid	C—H
2350	Lipid	C—H

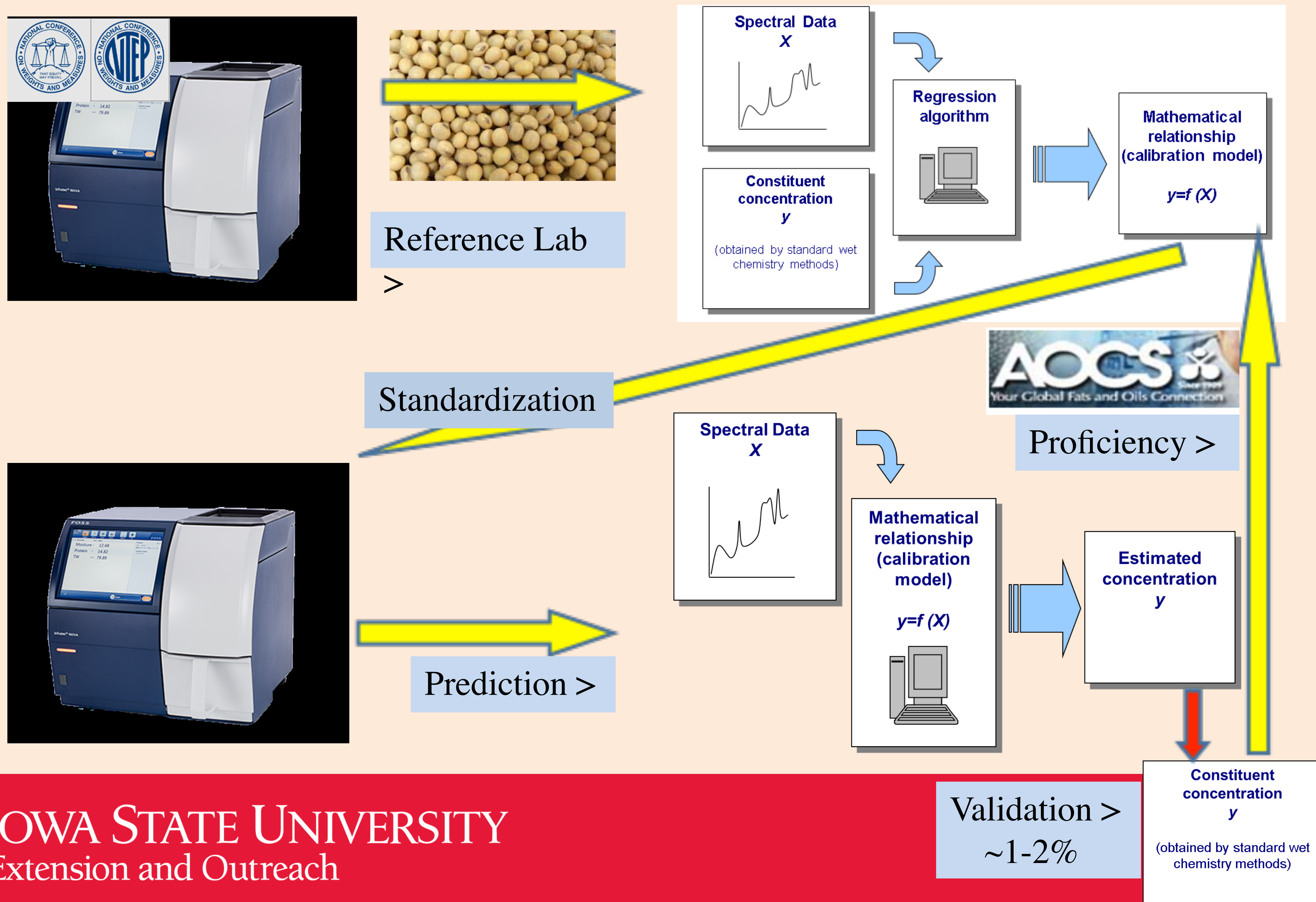
$$\hat{Y} = b_0 + b_1 x_1 + b_2 x_2 + \ldots + b_k x_k$$

k = 10 – 2000

Major components (> 2%) large effects

Subcomponents: (<2%) smaller effects

NIRS Operation Procedure



First, what is the goal?

Accuracy Needs

- Classification/selection 2-3
  - Accept – reject, ranking

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- Market Information 3-5
  - Guidance, relative comparisons, databases
- Trade, specification, refereed science 5+
  - Payments, decisions, citable, future work

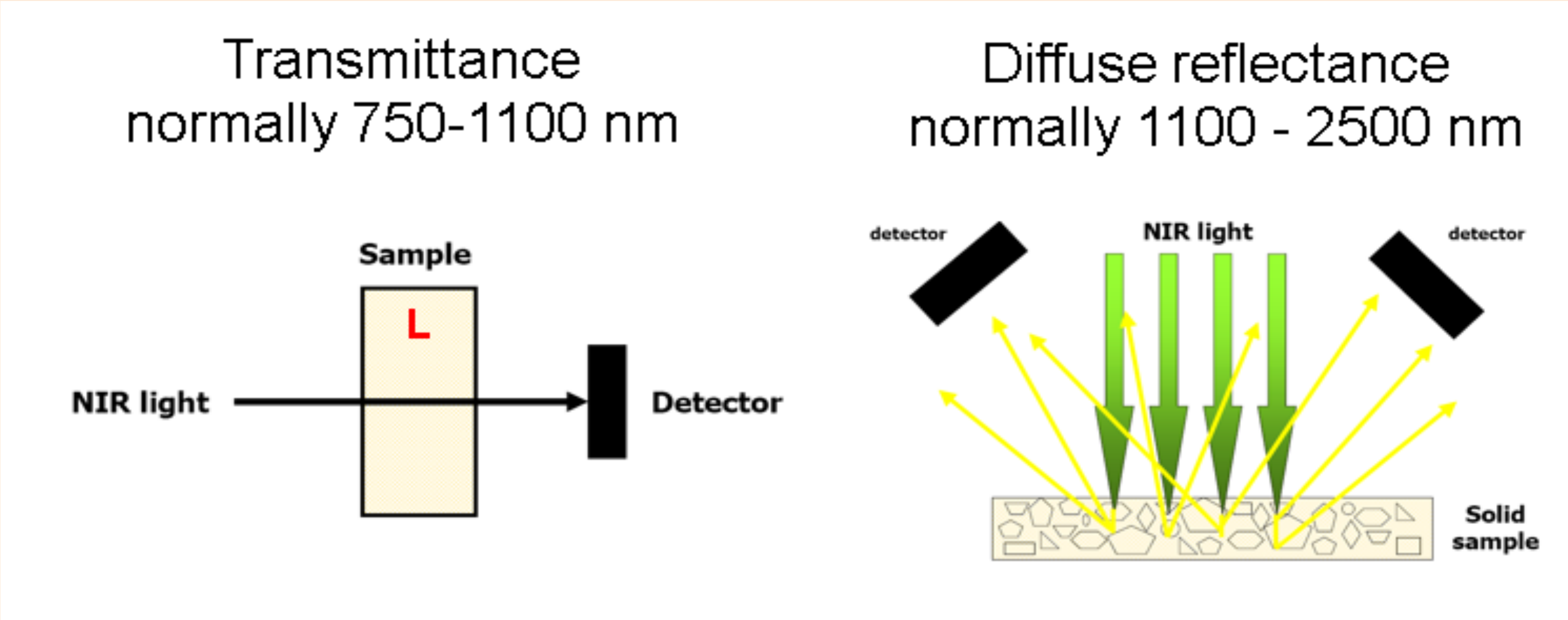
Know how much variability will be acceptable.

Accuracy in terms of RPD (std. dev. of data/std error of acc.)

Next, instrument choice

- How much time do you have per test?
- Could you grind, or not? Destructive, slow but more accurate for reflectance units.

- What technology would work best for each case?



NIRS Transmission-Based Units

Bruins OmegAnalyzerG

NIRS Reflectance-Based Units

Next: Where are you in the operation chain?

- Are you going to accept a calibration that you get from somewhere? Or do your own? What specific calibration is it?
  - Manufacturer
  - Other developer?
  - Calibration version/identification; scientific publication?
  - Update/monitoring protocol
- Are you going to standardize your own instrument? Or accept the standardization settings you are given?
- What is the reference basis of the calibration? Is that widely accepted in your market/application?
- Validation is totally your responsibility
  - Proficiency program?
  - Quality management program?

Measurement Statistics

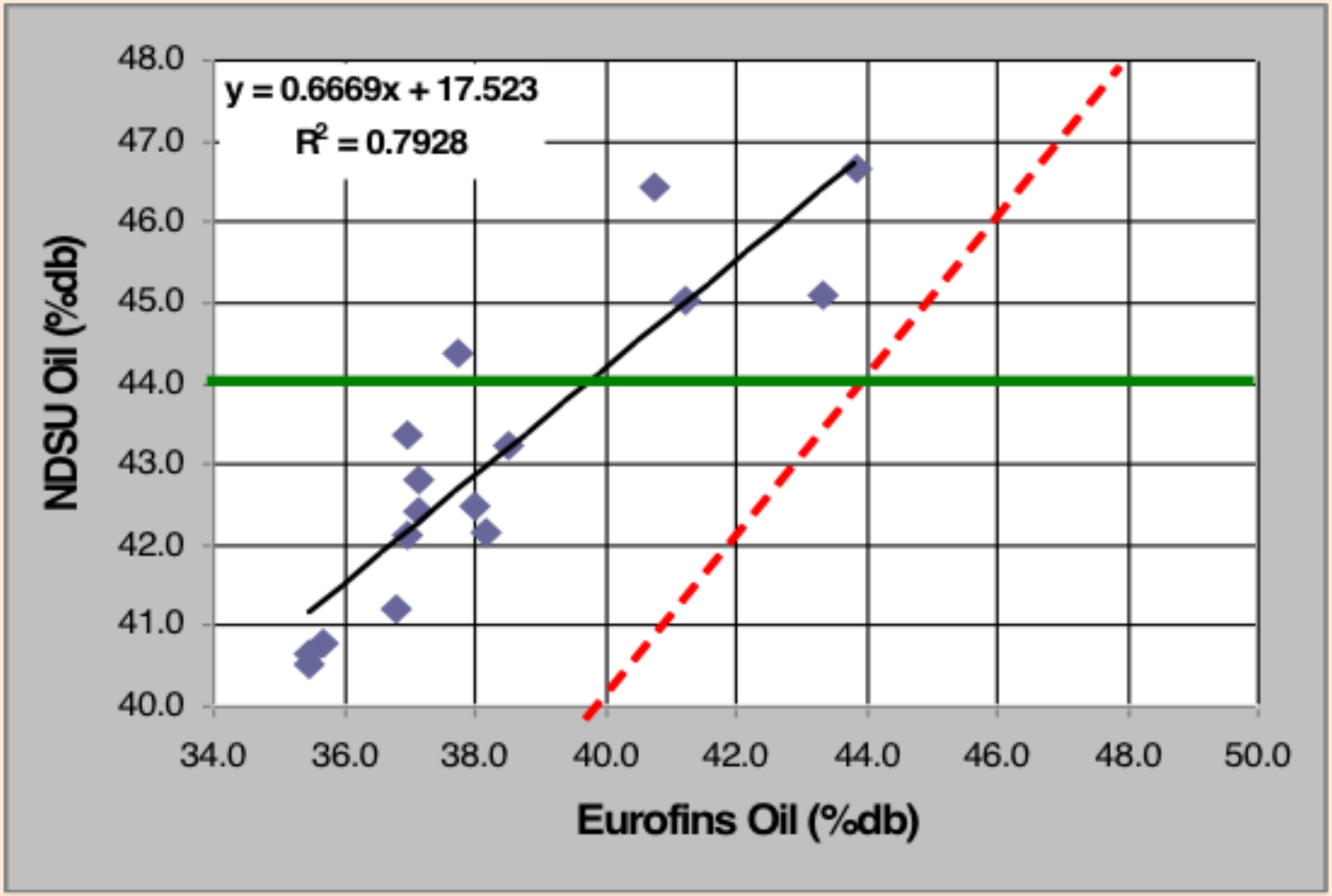
- **Repeatibility (Precision)**
  - Standard deviation across repeat tests

    Instrument construction
- **Reproducibility – within model (aka. Standardization)**
  - Standard deviation across like units

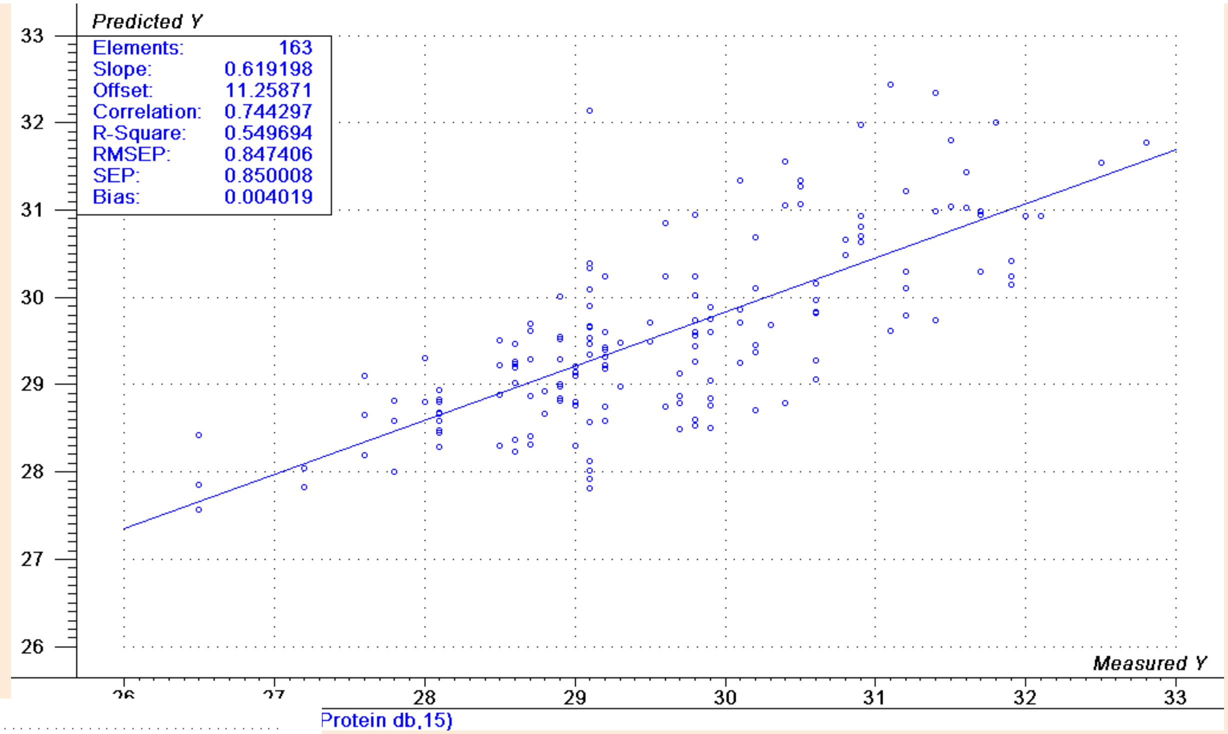
    Instrument uniformity
- **Equivalence - across models (aka. Harmonization)**
  - Standard deviation across all units

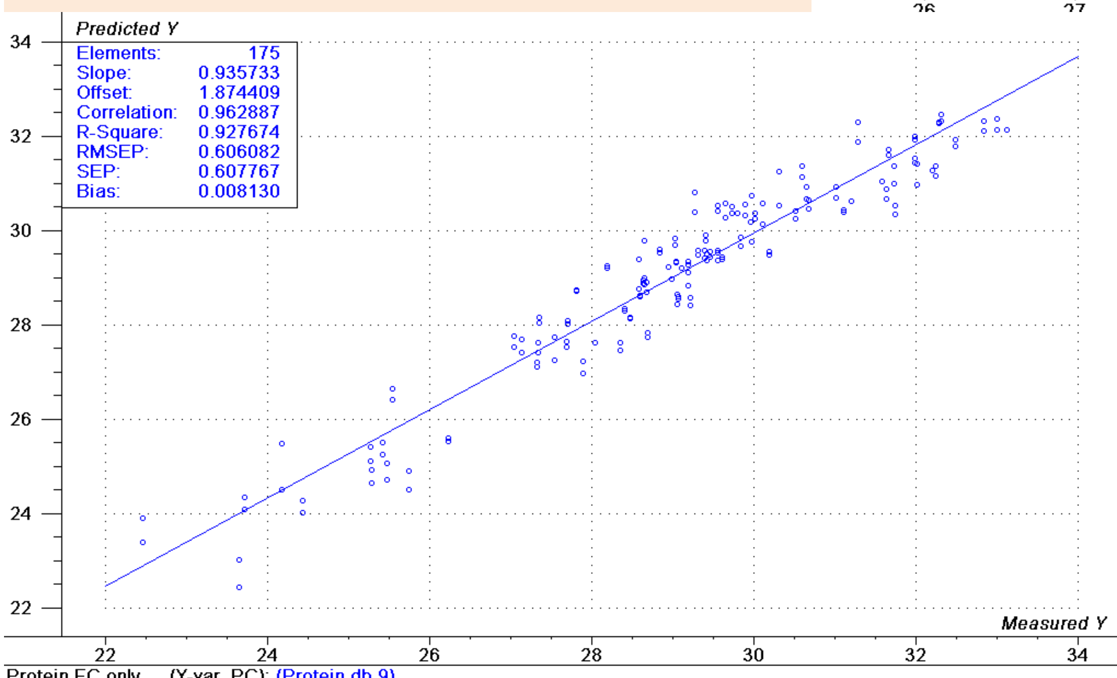
    System performance

Reference Method Differences
Oil in Canola



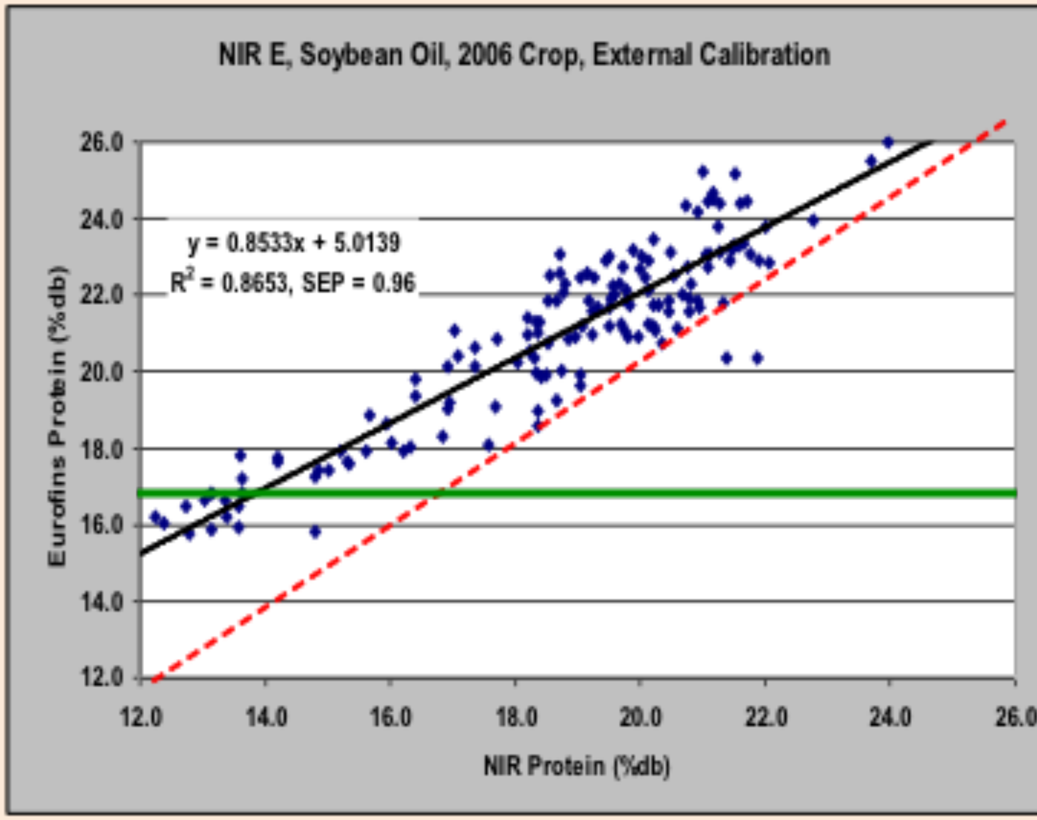
Reference Lab Performance Protein in DDGS



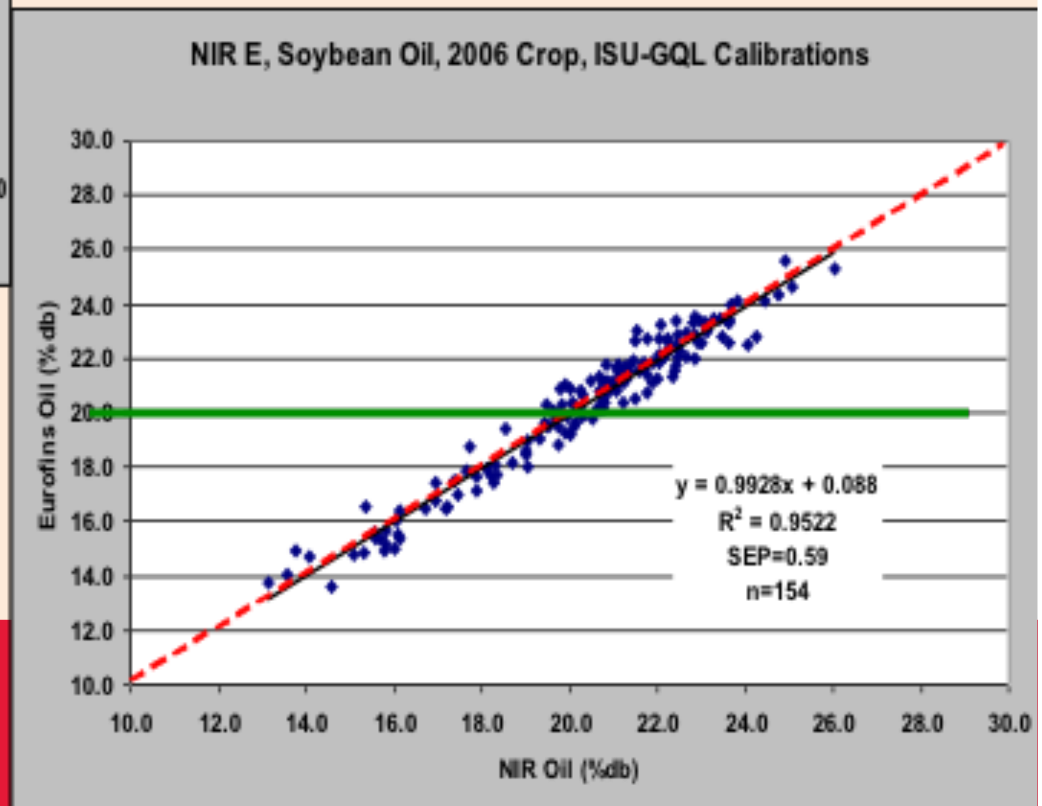


Same instrument and spectra, different lab values

Precalibrated Units



Same instrument and spectra, different calibrations



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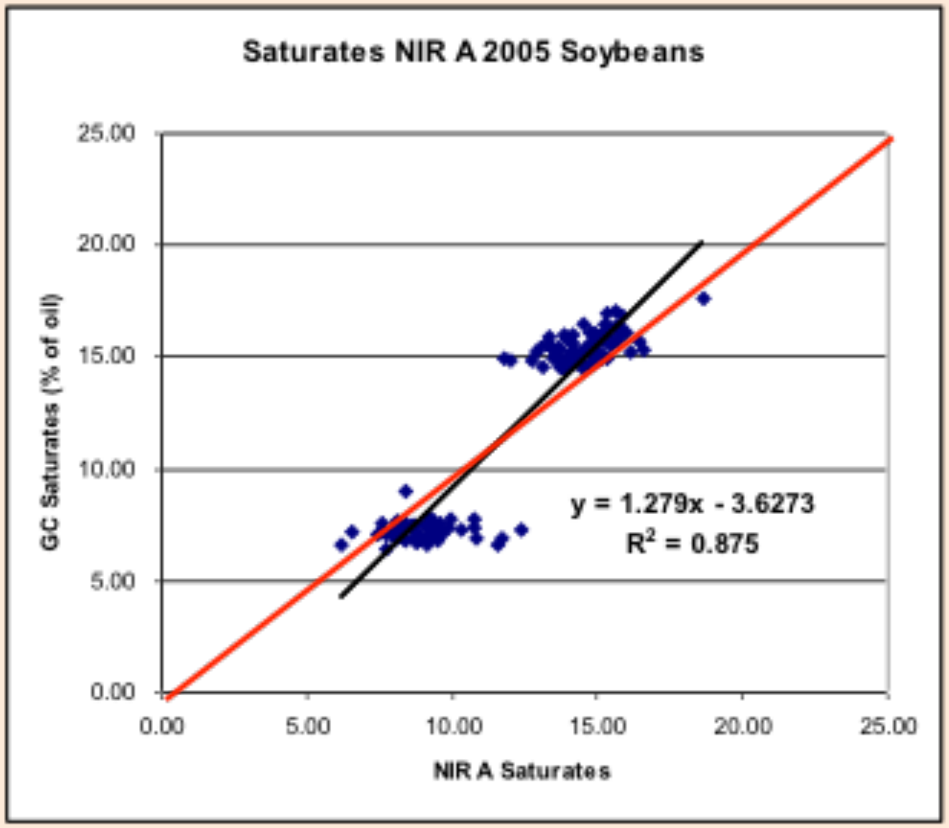
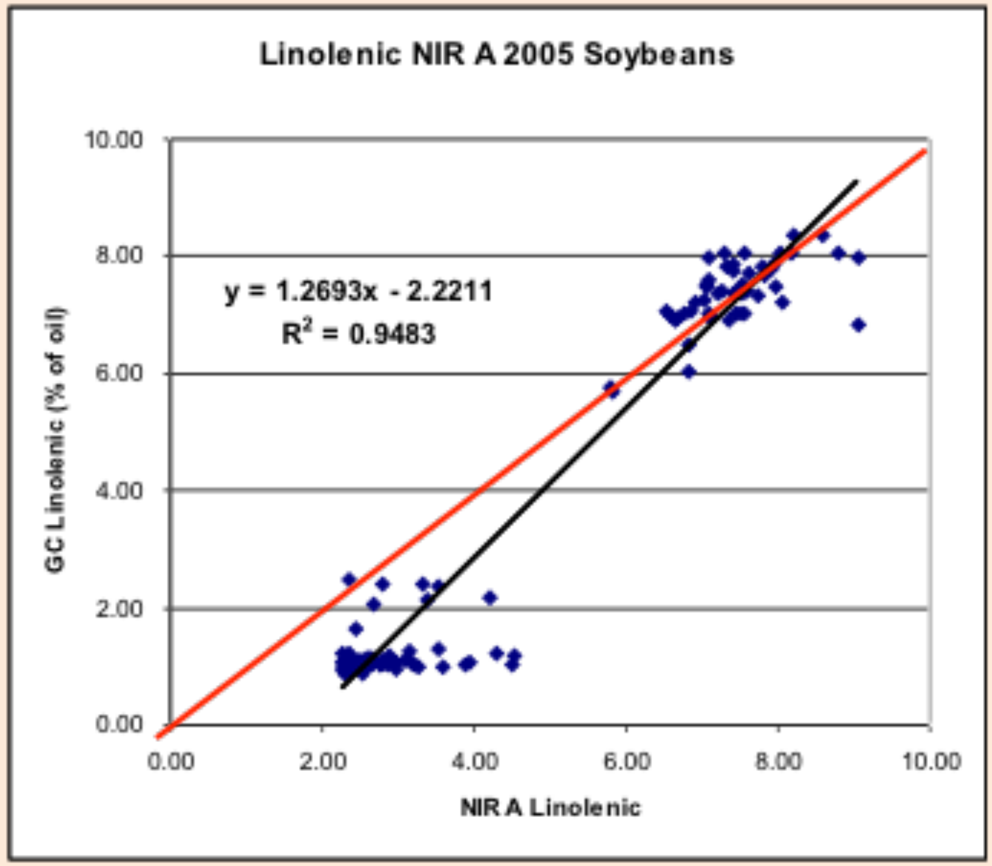
Validation – Classification

No difference within classes; classes separated



SEP = 1.30



SEP = 0.75

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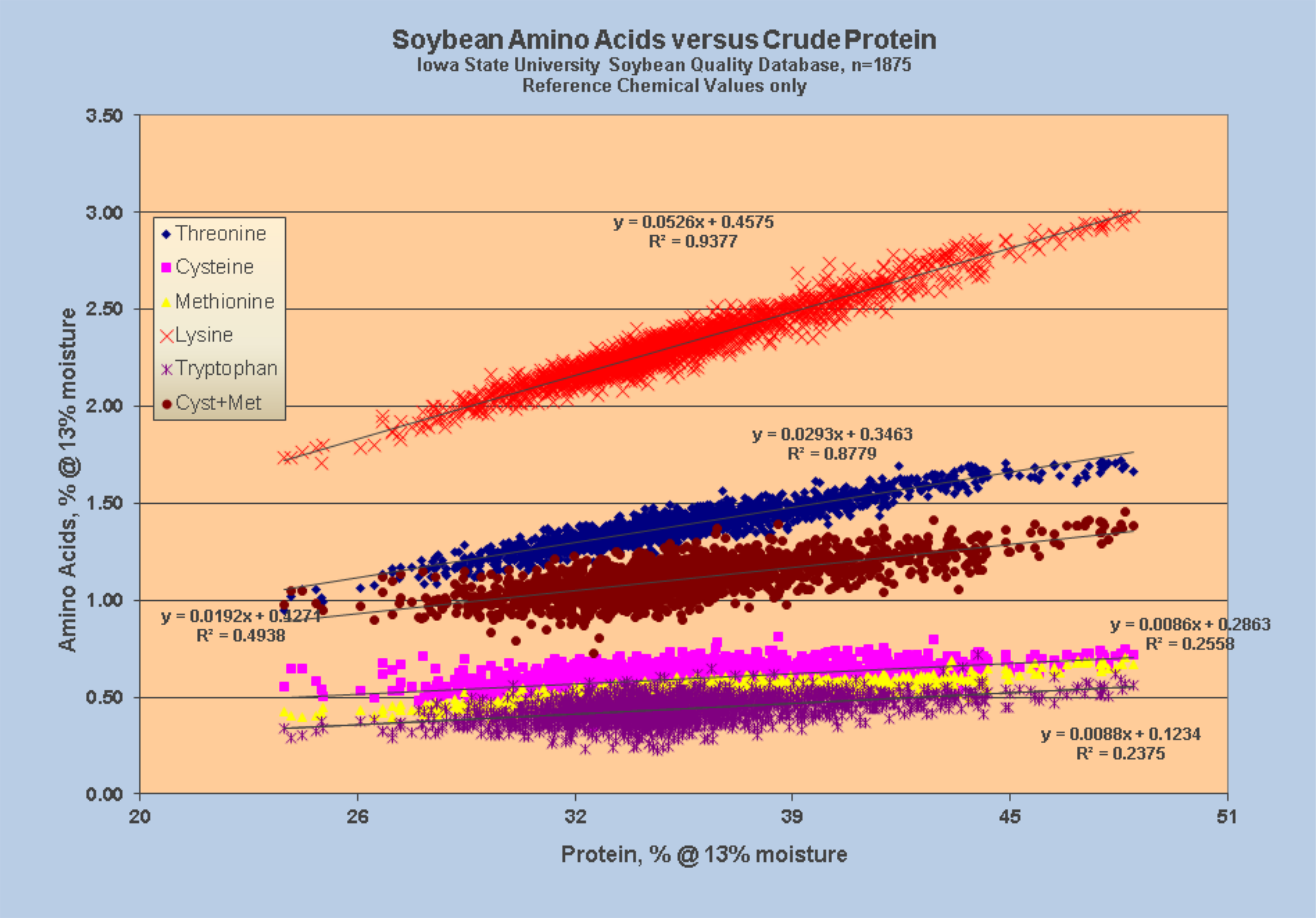

Iowa State University
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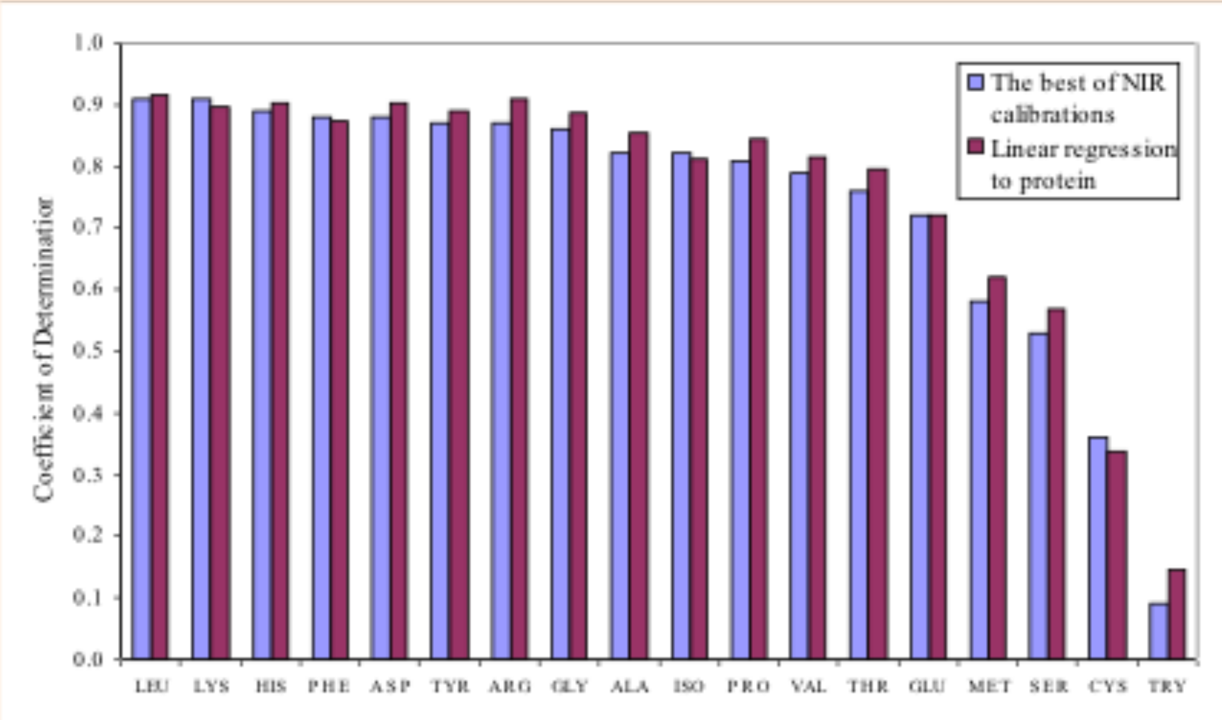
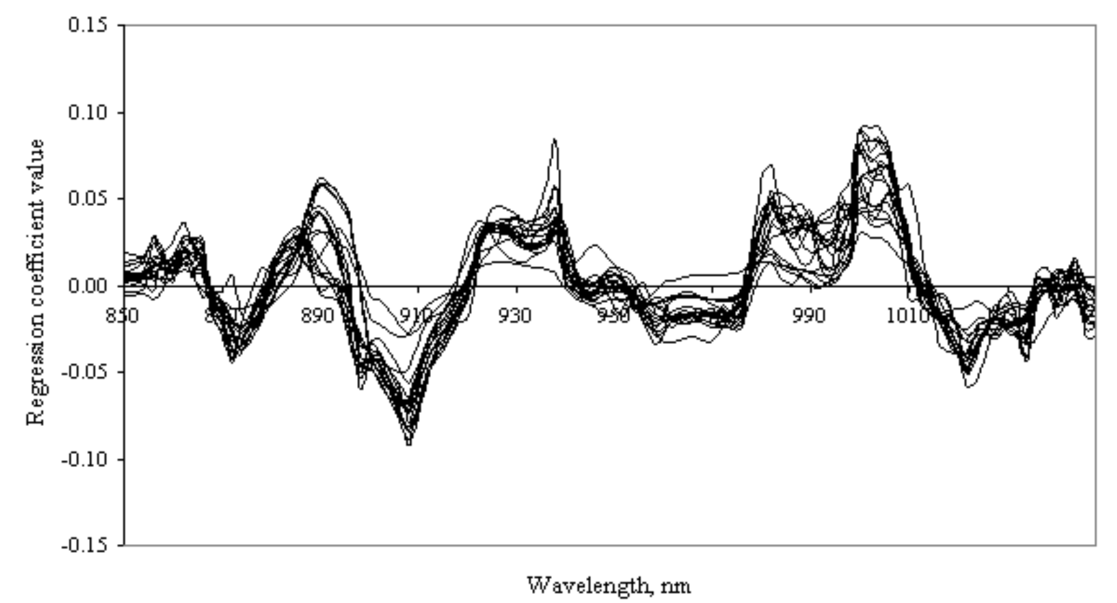
Correlated Y: Amino Acids



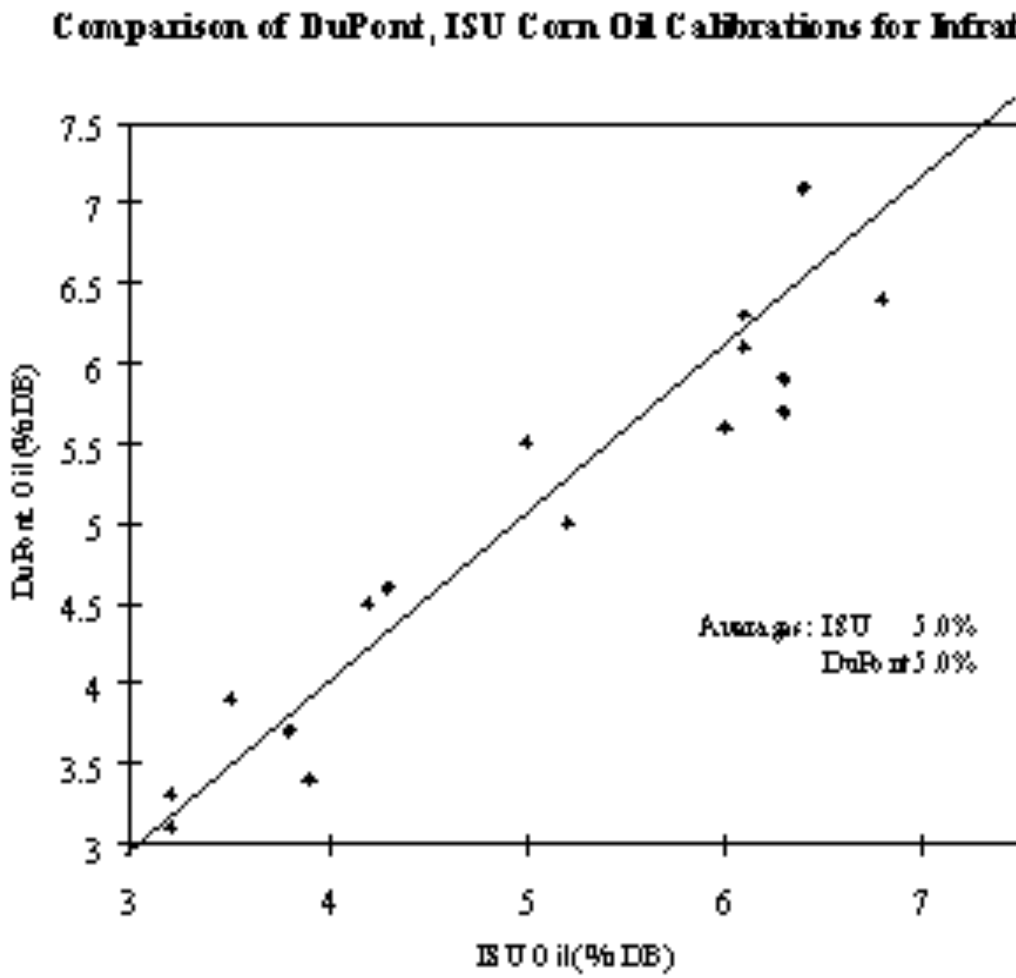
Comparison of NIR calibration models (average *r*2) with linear regressions of reference amino acids to reference protein

Regression vectors of 18 AA PLS models for FOSS Infratec 1241 Grain Analyzer; most of the curves follow the same pattern, which indicates that calibrations predict one constituent.

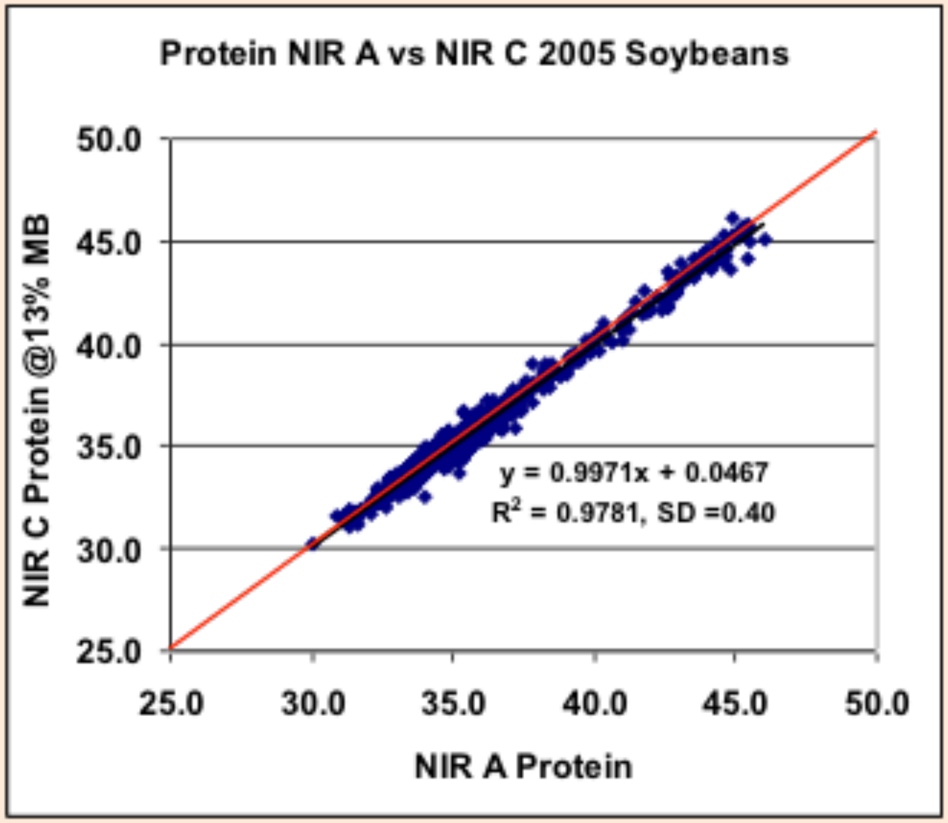


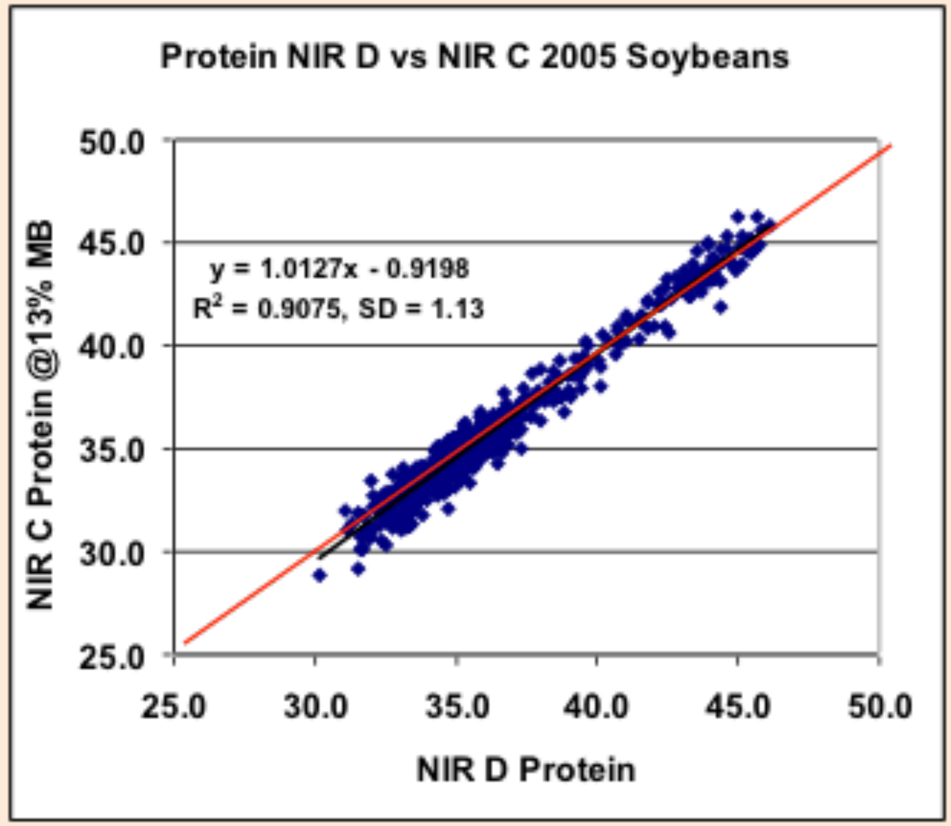
Platform Differences - Equivalence

DuPont, ISU Infratec Corn Oil Calibrations Comparison



Equivalence among well calibrated units





Similar technology (transmittance)
SEP to reference =0.50,0.45

Reflectance vs. Transmittance
SEP to reference = 0.50, 0.75

It all depends on the goal

NIRS Proficiency Data for Soybeans

Mean	Range	Standard deviation Between-Laboratory (Equivalence)	Within-Laboratory (Reproducibility)
--Protein, percent dry basis -----------------			
39.81 (34.63 @13%M)	38.1 – 41.3	2.00	1.25
--Oil, percent dry basis----------------------			
21.32 (18.55 @13%M)	19.3-24.6	1.68	0.85

AOCS SQT program, 2008 – 2010, 39 labs, 65 samples

NIR Equivalency Study – Cooperative Agreement

- Initiated in 2014. GIPSA and Iowa State University (ISU)
- Limited to models with NTEP Certificates of Conformance
- Criteria on a common sample set
  - 1) Precision and 2) SEP are acceptable
  - ucibility be ithin a

Perten IM9500

Bruins OmegAnalyzerG

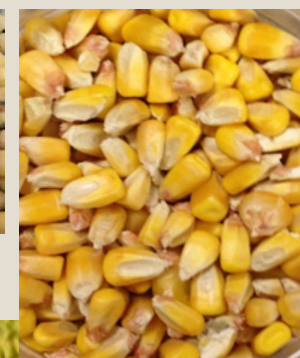
FOSS Infratec 1241

There were 5 copies of each instrument in the study

Study Description

	Std. (n)	Test (n)			
From GIPSA, GIPSA reference data					
Wheat	6	250	6 classes	Protein	12%MB
Barley	5	100	2 classes	Protein	DB (0%)
From Iowa State, Eurofins reference data					
Soybeans	20	145	1 class	Protein, Oil	13%MB
Corn	30	149	1 class	Protein, Oil	DB (0%)

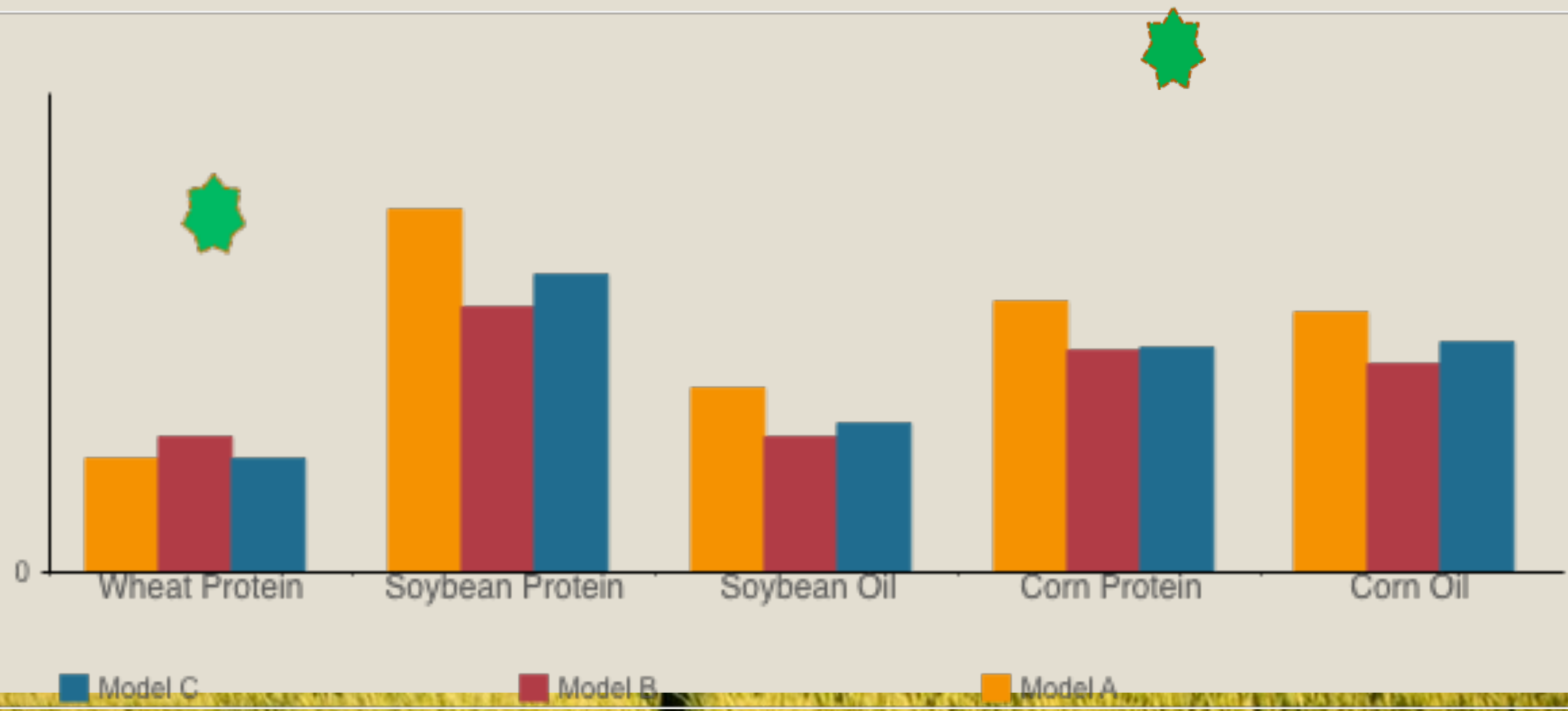
644*3*5*3reps
28,980 drops!



FOSS

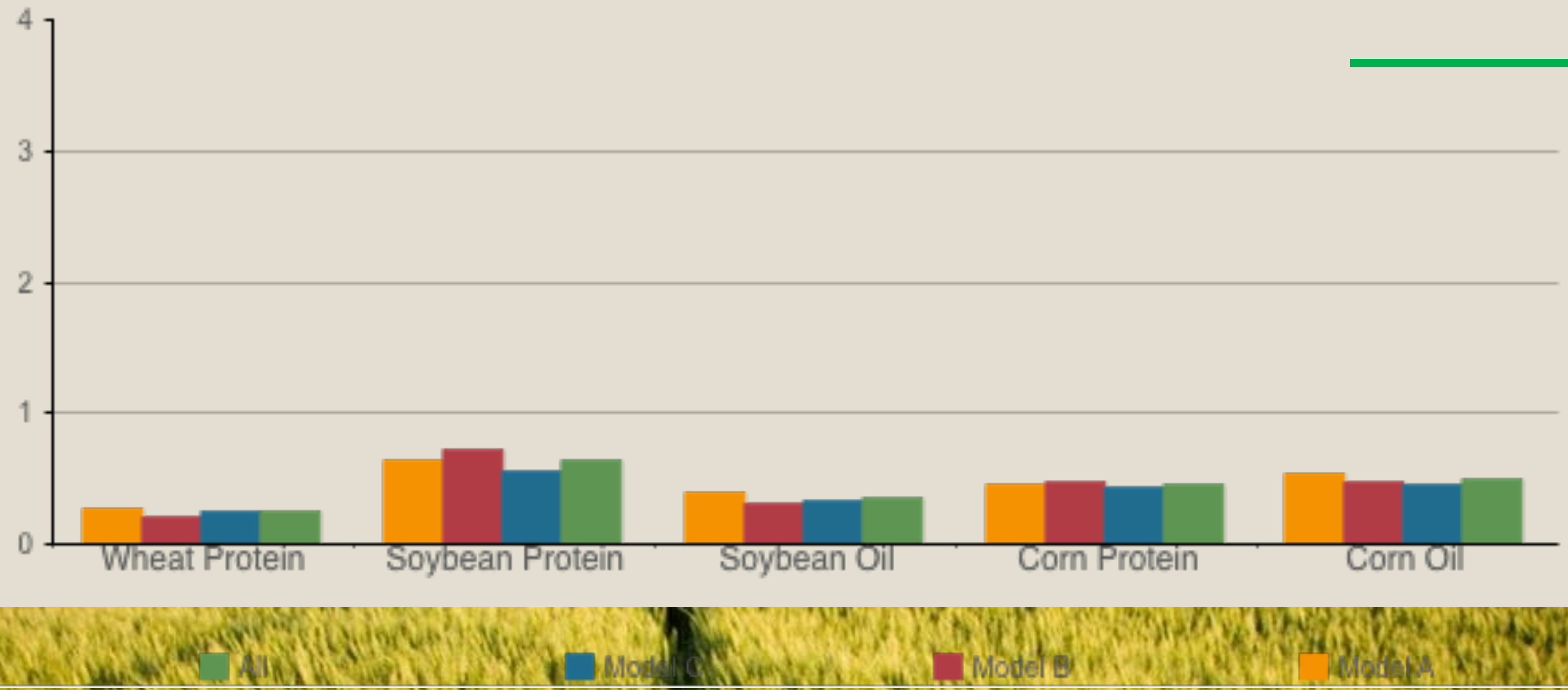
First Consideration

- Is the hardware (design) precise? **Yes**
  - **All meet NTEP Criteria**;



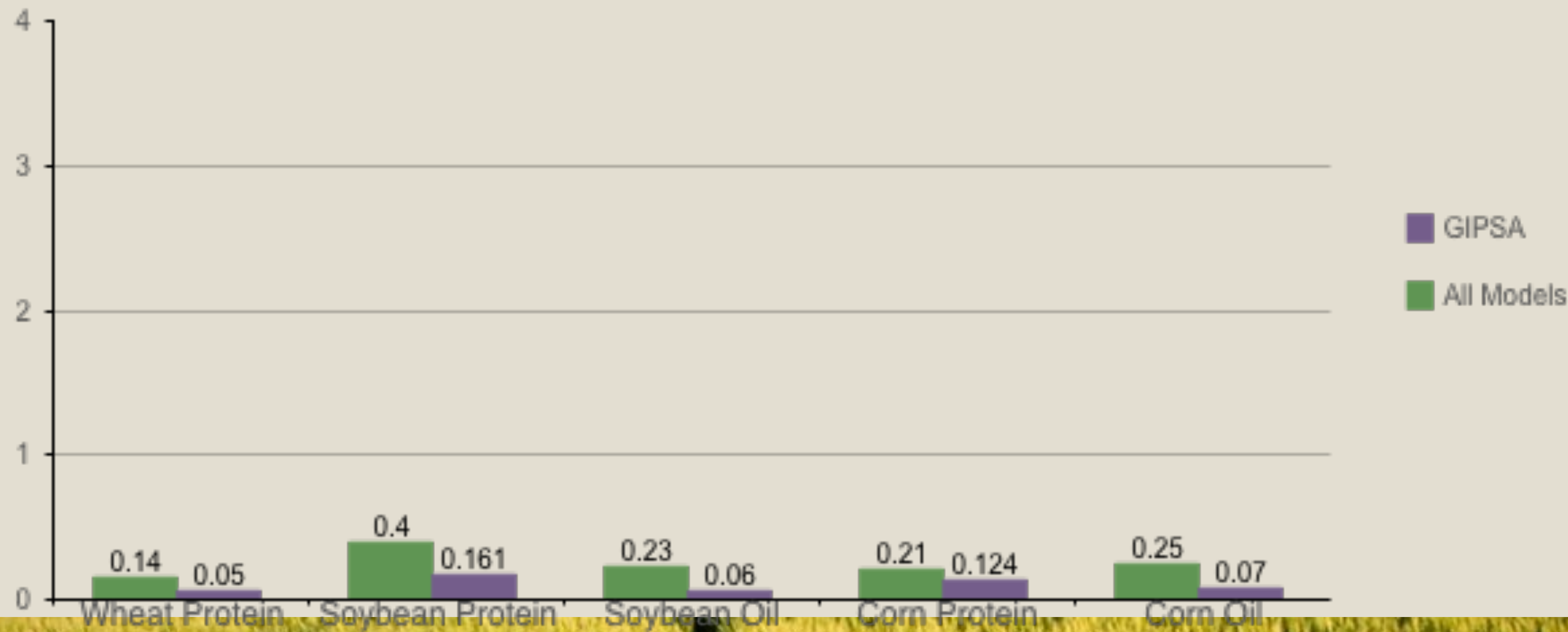
Second Consideration

- Are the calibrations accurate to the reference method?
  - **Close: Soybean protein could be improved by including newer varieties and more widely distributed results.**



Third Consideration

- Is the agreement within and between models (equivalence) acceptable? **Yes by NTEP**; **No by GIPSA/ISU definition**.



Strengthening (USB) NIRS Programs

1. Each user must understand their own goals and needs.

2. Each user must determine what instrument fits best

3. Each user must determine what their role(s) will be.

4. Establish approved, validated reference labs.
   - GIPSA for Official factors, or proven equality.
   - New factors: scientific review then lab approval.

Strengthening (USB) NIRS Programs

1. Expect scientific proof at each operation that others do.
   - The platform concept is critical: instrument+calibration
2. Participation in proficiency programs should be required
   - This will reduce equivalency issues.
   - Create a program for sb users; connect with GIPSA
3. Calibration/validation accuracy should be **published**.

4. Create Standards for inclusion of data in databases, public information.

International Diffuse Reflectance Conference 2018

July 29–August 2, 2018. Focus is on usage in practice.

Where To Find Us…

Iowa Grain Quality Initiative

Grain Quality LABORATORY

www.iowagrain.org

Analytical Programs
Quality Management Systems