ADSA 2025 Annual Meeting JUNE 22-25, 2025 • LOUISVILLE, KENTUCKY
#ADSA2025
Uniting the Dairy Science Community

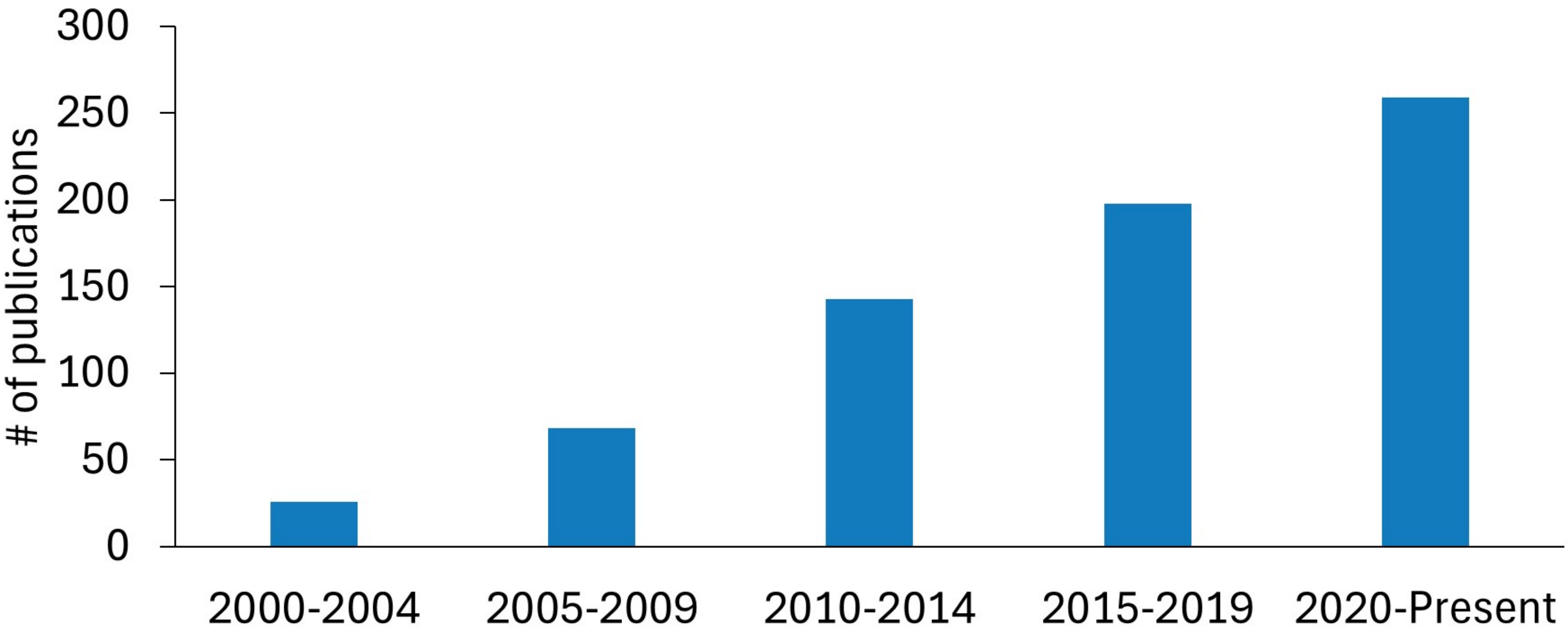
Assessing accuracy and validity of assays from commercial feed analysis laboratories

Nicole Schlau
R&D Manager, Dairyland Laboratories, Inc

NATIONAL ANIMAL NUTRITION PROGRAM

DAIRYLAND Laboratories, Inc.

Trends in Commercial Laboratory Use by JDS Studies



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Objectives

- Understand how a commercial laboratory:
  - Monitors and corrects systemic error
  - Identifies error in individual samples
- Understand how factors outside the lab affect interpretation of results
- Discuss contribution of NIR data to feed composition databases for atypical feeds
- Review examples to interpret outliers and distinguish error from true variation

Method matters!

- Empirical vs. rational methods (Ferreira and Thiex, 2022)
  - Results determined by a specific method (empirical)
  - Quantification of a specific analyte (rational)
- Most feed analyses are empirical
  - DM
  - Fiber: ADF, NDF
  - Fat: EE
  - Ash or organic matter
  - WSC, ESC

Monitoring systemic error

- Participation in proficiency testing programs
  - National Forage Testing Association (NFTA)
  - Association of American Feed Control Officials (AAFCO)

- Participation in ring test studies

  (Hristov et al., 2010, Hall and Mertens, 2012; many others)

- Internal quality control samples

Where does analytical error begin?

- Improper sampling technique

- Improper handling

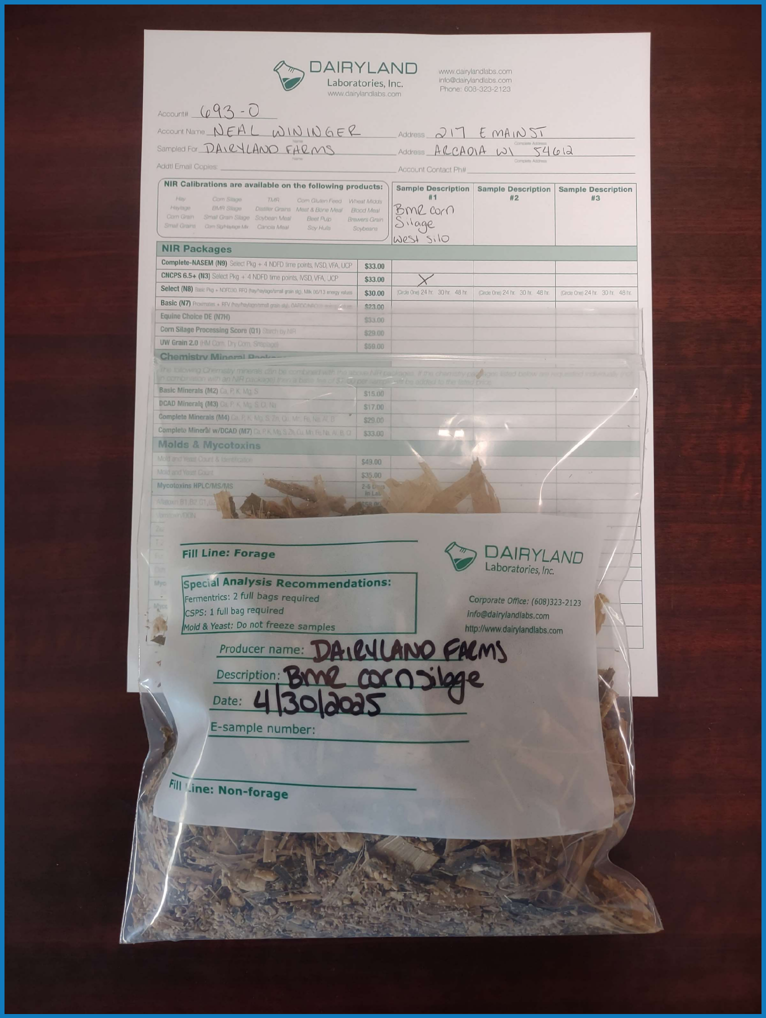
- Sample identification, classification and preparation

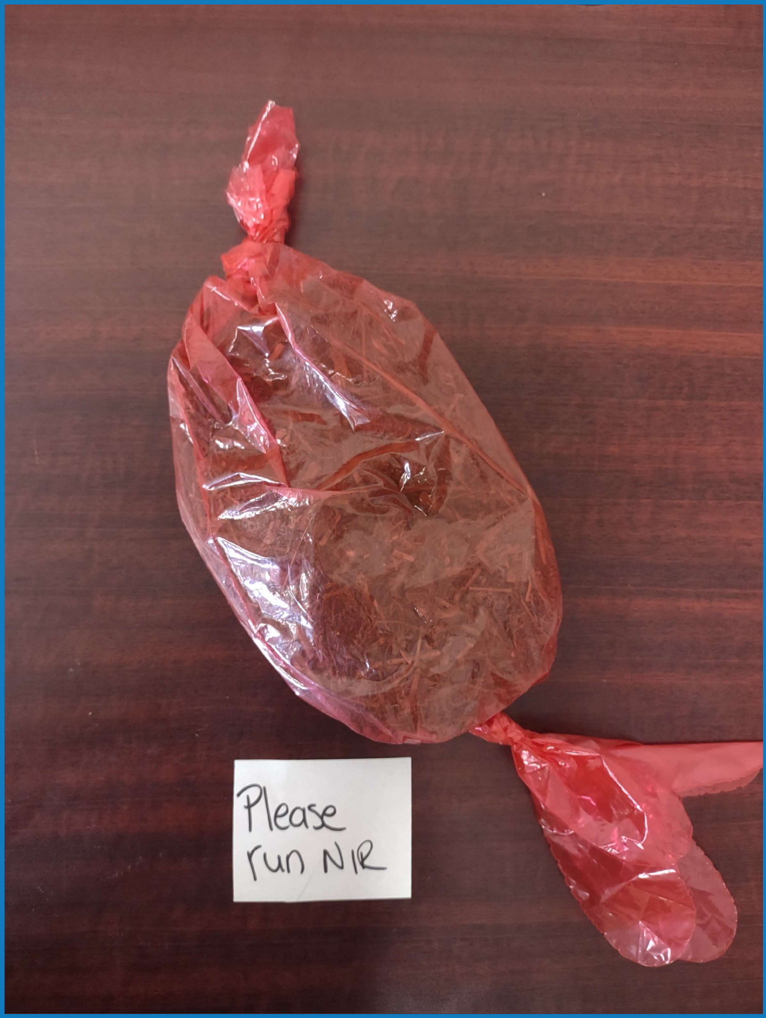
Role of NIR in Feed Analysis

- Near-infrared reflectance spectroscopy

- Secondary analytical measurement
  - Calibrated from chemistry

- ~90% of Dairyland Laboratories' database (2021-present) contains NIR data
  - Either alone or alongside chemistry packages (ex. minerals, digestibility)


DAIRYLAND
Laboratories, Inc.
NIR Packages
Fill Line: Forage
Special Analysis Recommendations:
Fermentrics: 2 full bags required
CSPS: 1 full bag required
Mold & Yeast: Do not freeze samples
Producer name: DAIRYLAND FARMS
Description: Bmr cornsilage
Date: 4/30/2025
E-sample number:
Fill Line: Non-forage


Please
run NIR

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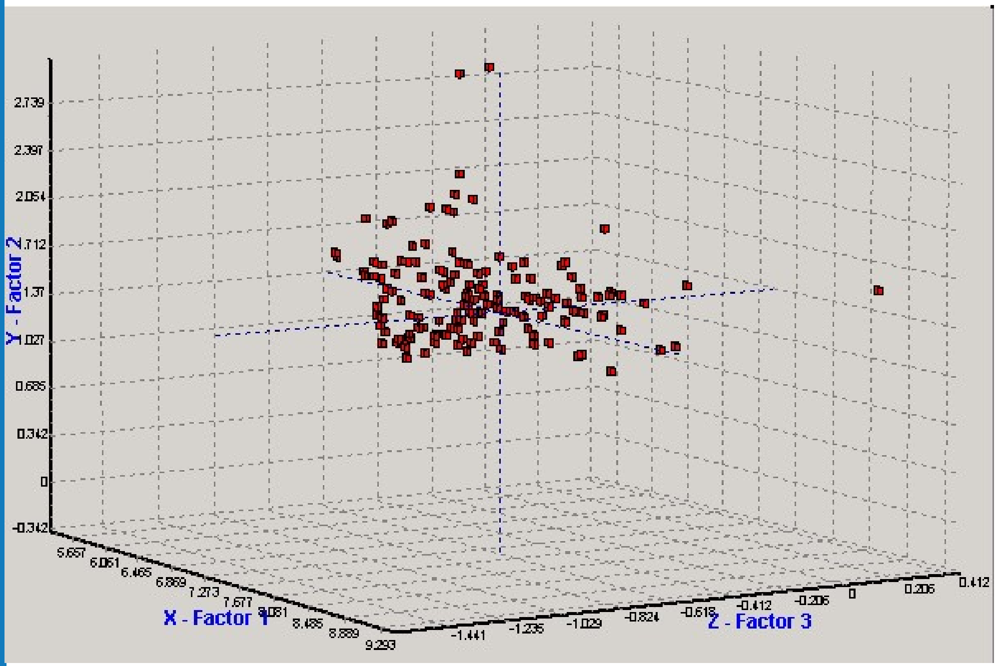
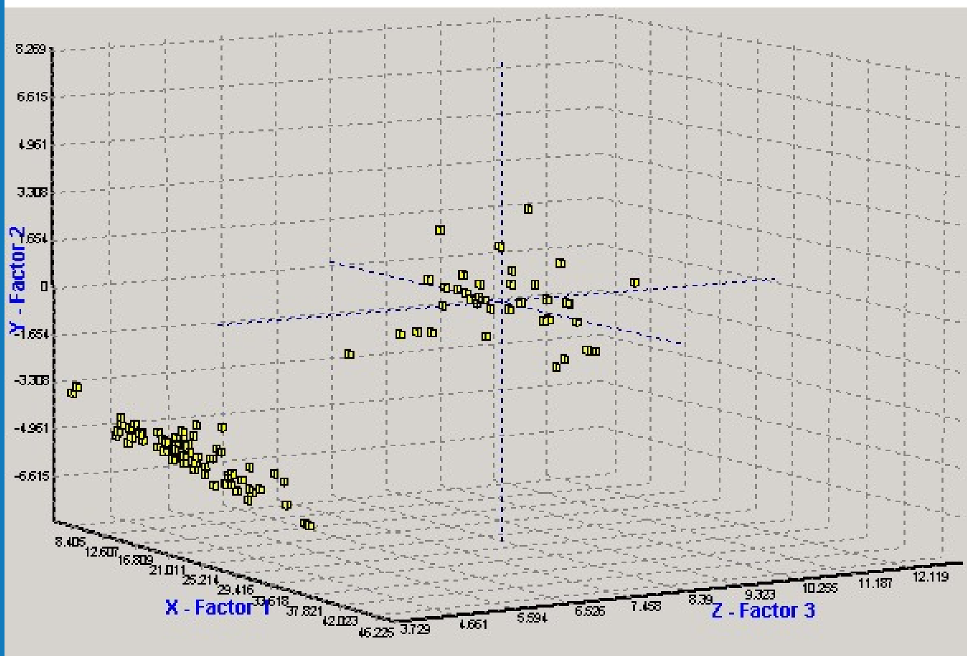
Sample identification: PCA of NIR Spectra



- Principal component analysis
- Summarizes variation in NIR spectra
- Global H value
  - Closer to 0 = close to center of cloud
  - > 3 may be outlier samples

How many different sample types are shown?

Sample identification: PCA of NIR Spectra



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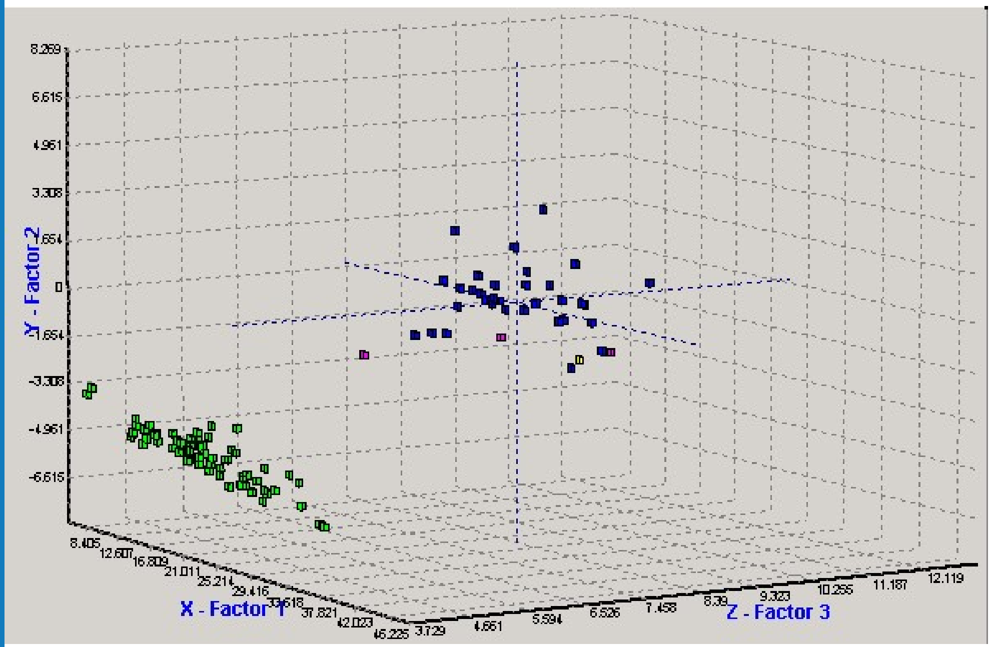

Distillers | Blood meal

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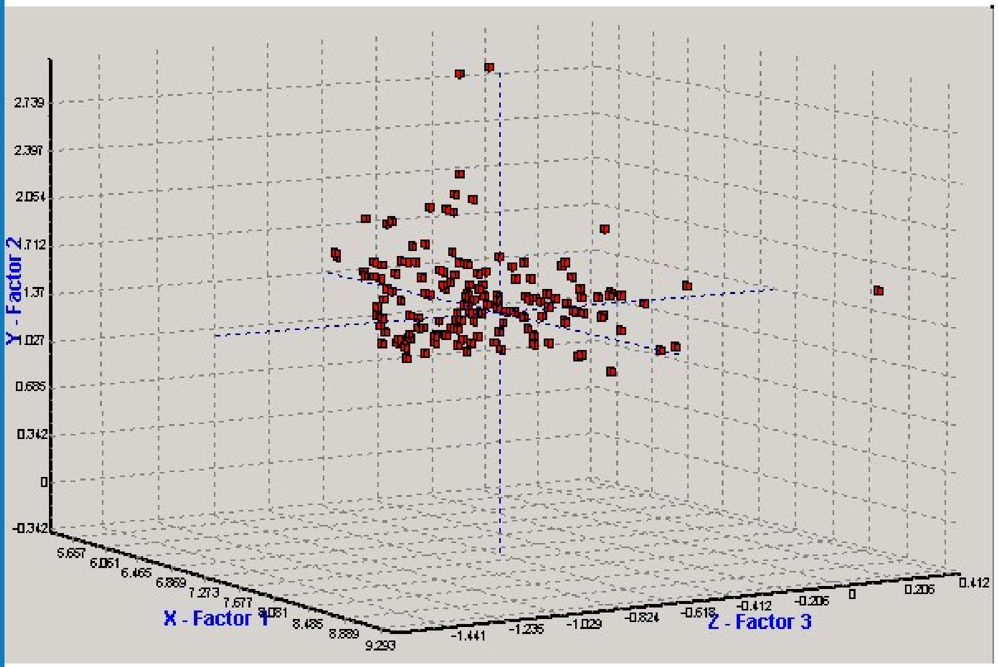

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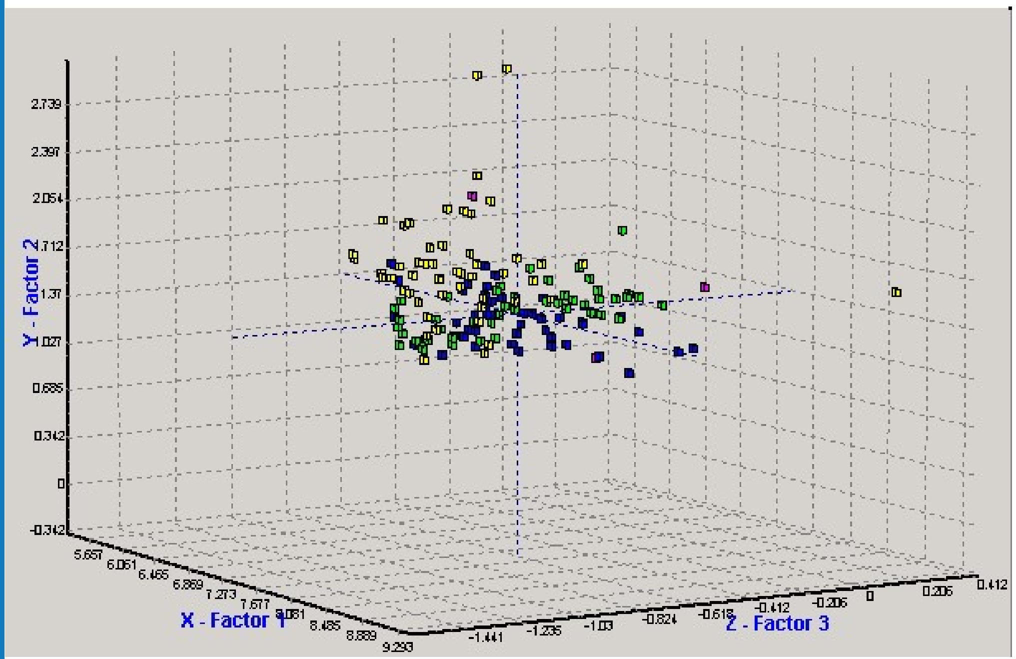


Alfalfa ■ Corn silage ■ Sorghum/sudan ■

- Spectral analysis may not be reliable for the diverse sample population in a commercial laboratory
- Advanced classification algorithms are only as good as the training data

Sample identification: PCA of NIR Spectra

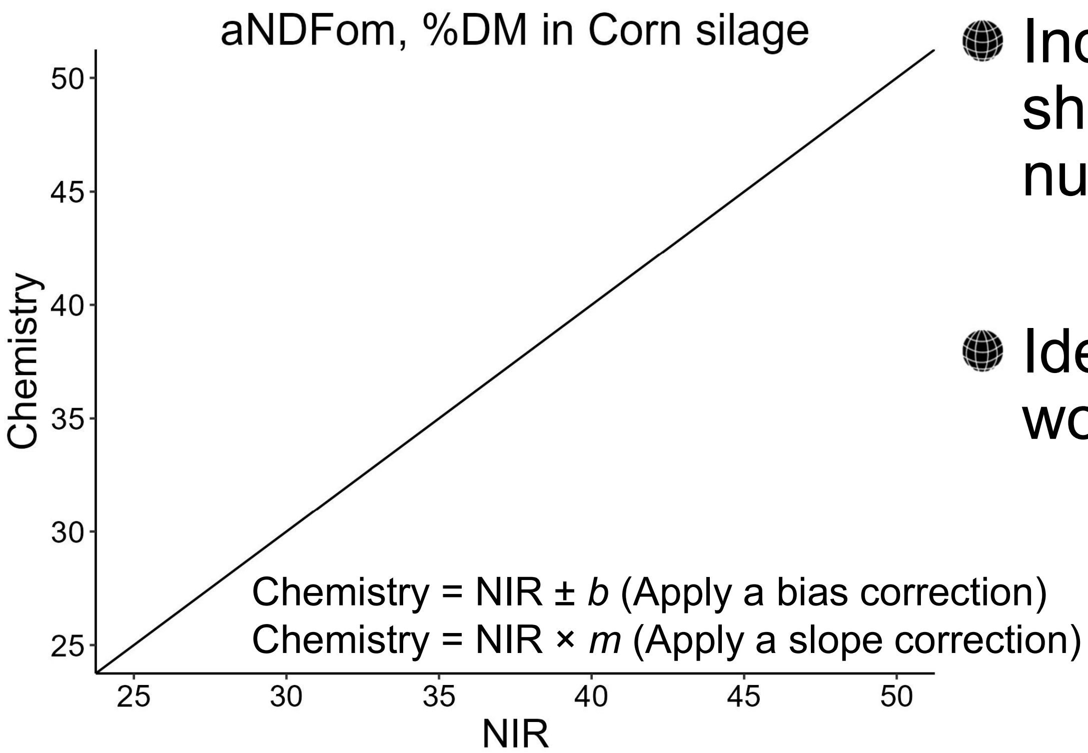
Standard error of prediction for mixed vs. crop specific calibrations

	Mixed small grain silages	Sorghum-Sudan	Mixed grass/legume	Alfalfa
CP	1.15	0.98	0.93	0.80*
ADF	1.92	1.63	1.45	1.47
aNDFom	2.54	1.40*	1.41	1.29+

*Values differ significantly ($P < 0.05$), Values tended to differ $P < 0.10$

Schlau et al. 2025

NIR Validation

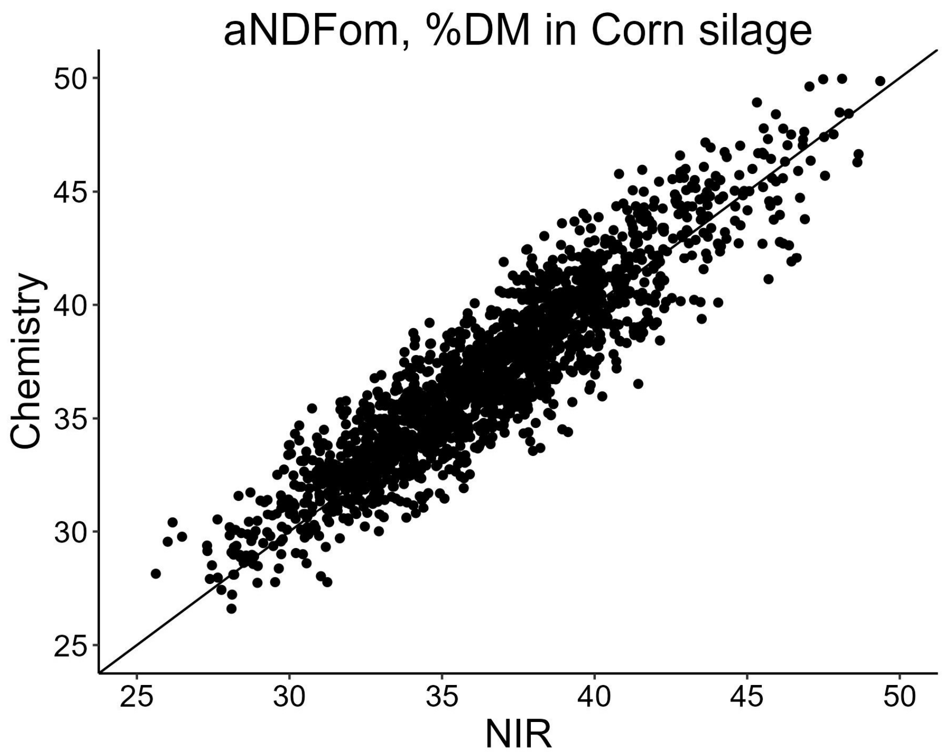


- Independent validation set should cover normal range of nutrient
- Ideally every measured value would fall on the y = x line

$$\text{Bias} = \text{Mean}_{NIR} - \text{Mean}_{Chemistry}$$

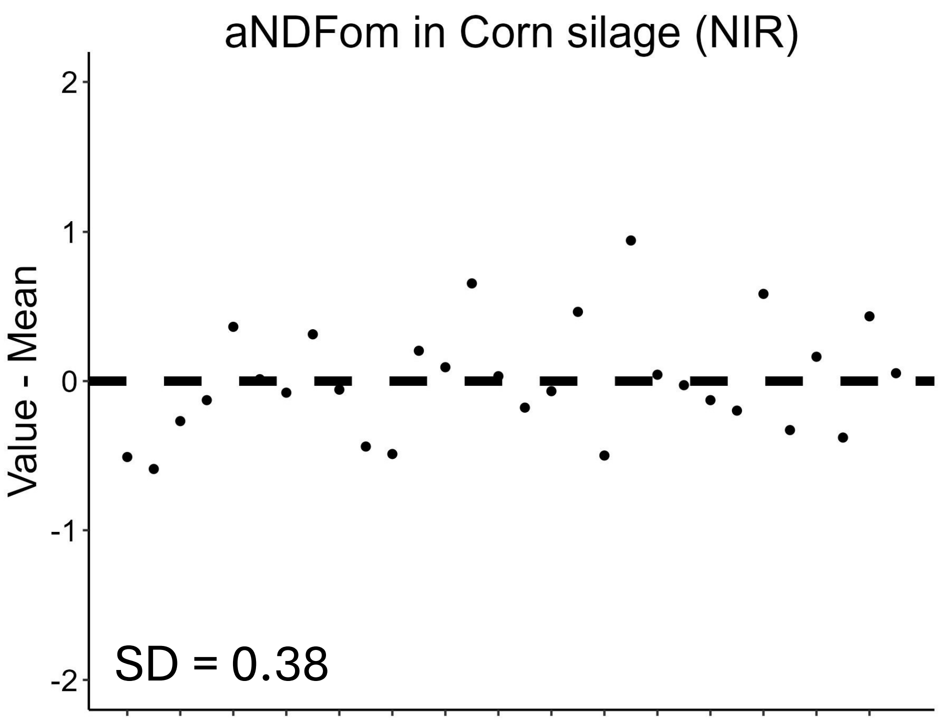
$$\text{Slope} = \frac{\Delta \text{Chemistry}}{\Delta \text{NIR}}$$

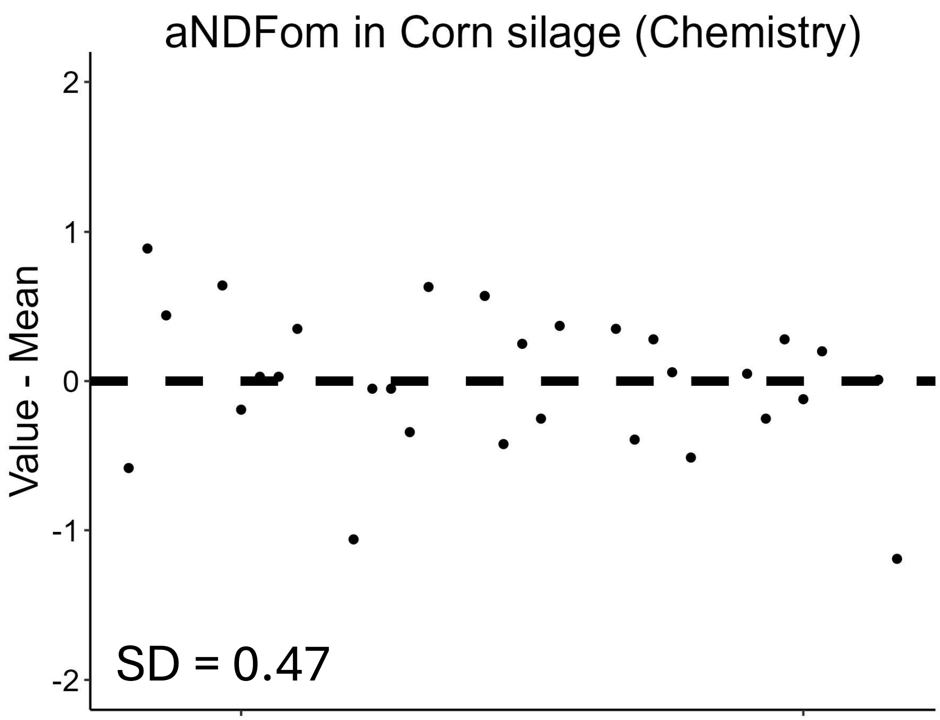
NIR Validation



Slope	0.94
SEP (RMSE)	1.69
Bias	0.43
R^2	0.84

What is normal variation?: Corn silage



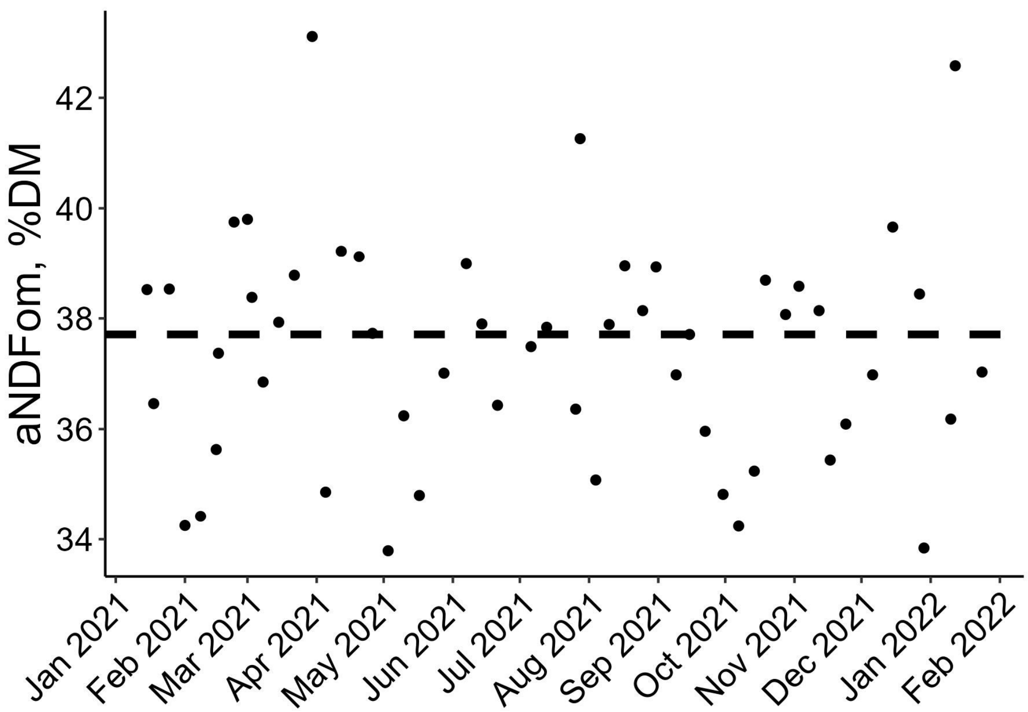


What is normal variation?: Corn silage

- SEP ≈ 1.5 – 2x the variation of the chemistry method

Corn silage check sample Dairyland	Corn silage Mertens (2002)
Mean: 33.27	Mean: 36.29
SD: 0.47	s_r: 0.60
n = 30	n = 12

What is normal variation?: Corn silage



Range of 9.32 units, SD = 2.05

- Sampling error accounts for ~35% of variation for corn silage NDF
  St-Pierre and Weiss, 2015
- Frequent sampling helps separate true variability from sampling error
  Weiss and Tebbe, 2020
- NIR is precise, economical and rapid

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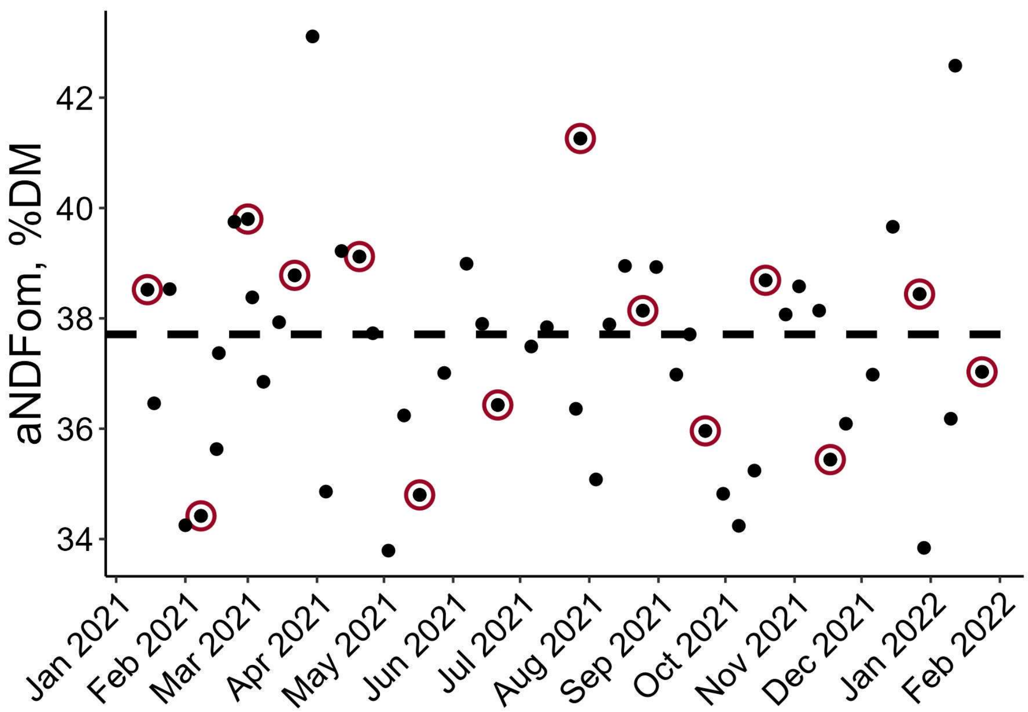
What is normal variation?: Corn silage



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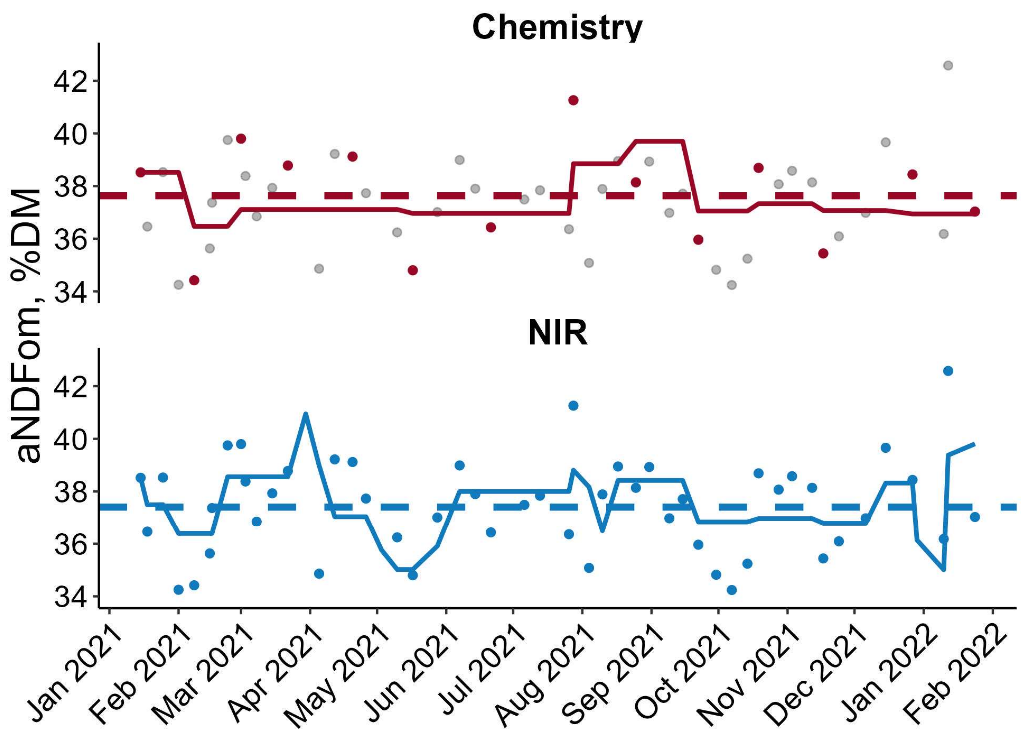

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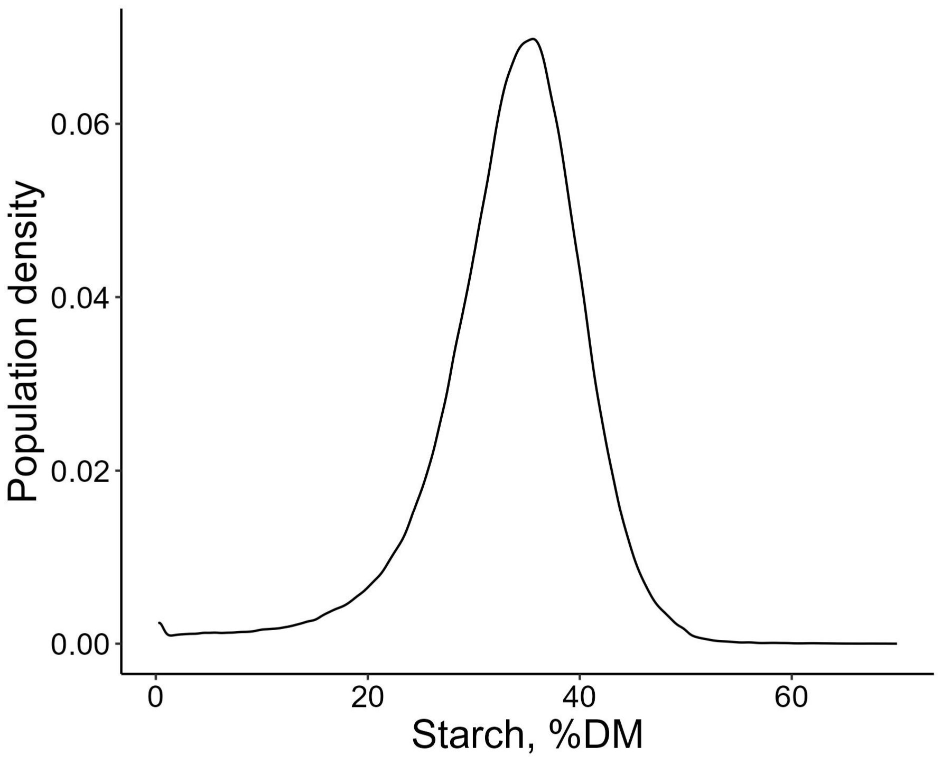
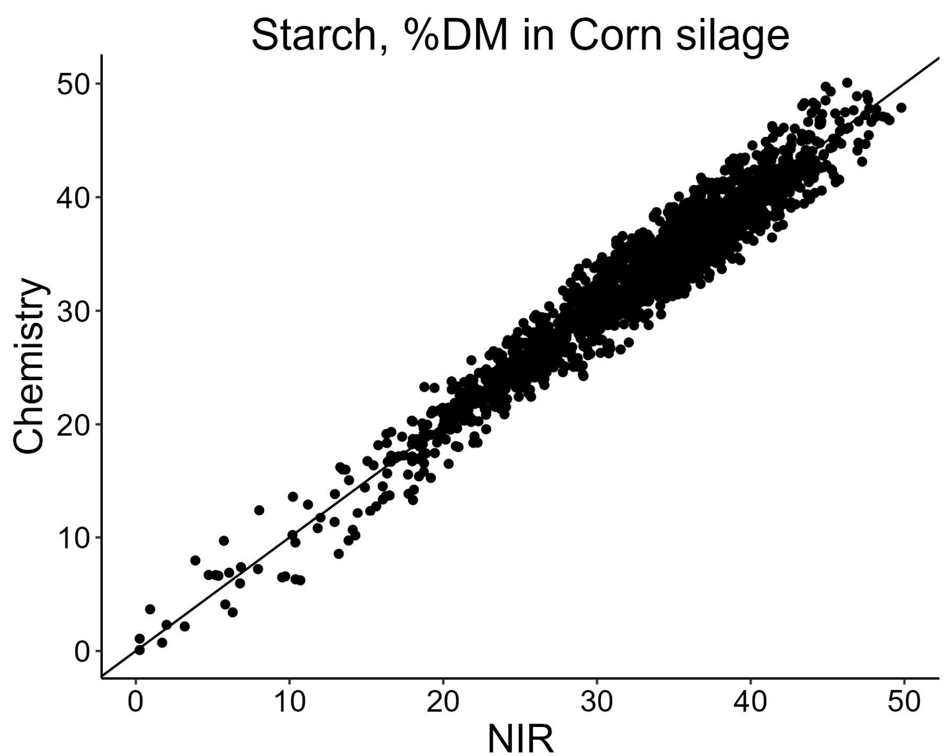
Predicting atypical feed by NIR

- NIR is not appropriate for every situation
  - Significant soil contamination, some research trials
- NIR calibrations cover the expected range of a feed type
- SEP is similar for typical and atypical samples, provided the samples are correctly identified
- Using NIR data in feed databases requires more samples to account for prediction error

Predicting feed anomalies by NIR





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Behind the Report: NIR & Chemistry

Sample Check-in → **Sample Preparation**

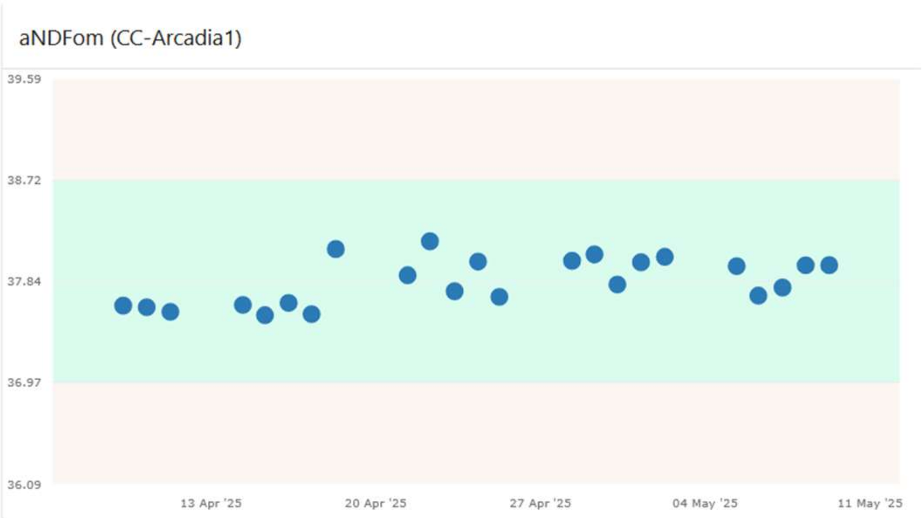
1-mm abrasion mill

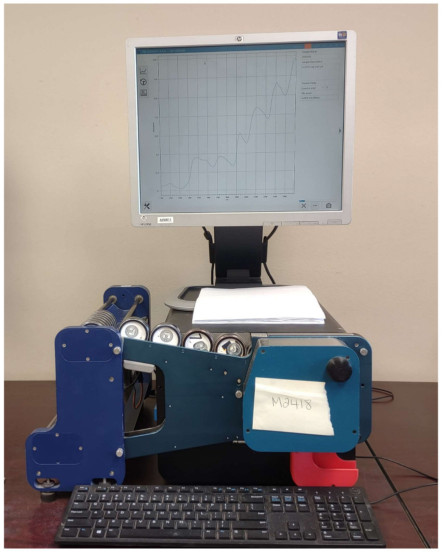
Subsample

Oven dry

Behind the Report: NIR & Chemistry

Sample Check-in → **Sample Preparation** → **Collect NIR Spectra**





Behind the Report: NIR

Sample Check-in → Sample Preparation → Collect NIR Spectra → Quality Control

- Verify sample was dried sufficiently
- Is the sample type accurate?
- Are there reasons to recommend chemistry?
  - High ash
  - Severe nutrient discrepancies (ex. negative predictions or predictions > 100)

Behind the Report: NIR

Sample Check-in → **Sample Preparation** → **Collect NIR Spectra** → **Quality Control** → **Report!**

DAIRYLAND
Laboratories, Inc.

Dairyland Example Account
213 Main St.
Arcadia, WI 54612

Sample Date: 2024-12-10
Sample No.: 008-2412-1759502

Account No.: 693 (0)
Sampled By: Your Feed Dealership
Sampled For: DOUG HARLAND
Product: Bunk 2

Test Mode: N3
Feed Type: Whole plant corn
Sub Type: Conventional

Moisture	64.54%		
Dry Matter	35.46%		
pH	3.54		

		Dry Basis	90% Range*
Crude Protein	%DM	7.15	5.80 - 9.00
AD-ICP	%DM	0.10	0.48 - 1.08
ND-ICP w/SS	%DM	0.40	0.73 - 2.42

18:2 Linoleic	%TFA	52.22	
18:3 Linolenic	%TFA	5.42	
Ash	%DM	4.24	2.81 - 6.19
Calcium	%DM	0.18	0.16 - 0.31
Phosphorus	%DM	0.23	0.19 - 0.28
Magnesium	%DM	0.11	0.13 - 0.23
Potassium	%DM	1.08	0.77 - 1.35
Sulfur	%DM	0.10	0.08 - 0.13

Behind the Report: Chemistry

Sample Check-in → Sample Preparation → Collect NIR Spectra → Chemistry Analyses → Quality Control → Report!

- Verify instrument diagnostics pass
- Verify check sample passes (each chemistry test)
- Verify results don’t go over high standard (if applicable)
- Compare results to NIR (if applicable)
  - Verify sample was dried sufficiently
  - Is the sample type accurate?
  - Do results deviate from NIR?
    - Recheck chemistry
- Severe nutrient discrepancies
  - Recheck chemistry

Is it weird or is it wrong?

Moisture	76.60%		
Dry Matter	23.40%		
pH	6.10		
		Dry Basis	**90% Range***
Crude Protein	%DM	18.26	16.9 - 24.6
AD-ICP	%CP	9.97	4.62 - 12.6
ND-ICP w/SS	%CP	8.32	
Protein Sol.	%CP	53.34	37.8 - 67.5
Ammonia-CP	%CP	77.56	0.00 - 28.3
Total Amino Acids	%DM	18.06	11.1 - 18.3
Total Amino Acids	%CP	98.90	63.1 - 82.4
Lysine	%CP	5.64	3.12 - 4.80
Methionine	%CP	1.81	1.20 - 1.80
Isoleucine	%CP	5.20	2.82 - 3.76
Leucine	%CP	9.97	5.05 - 6.97
Histidine	%CP	2.08	1.21 - 1.70
ADF	%DM	34.40	27.2 - 40.4
aNDF	%DM	41.78	33.0 - 47.8
aNDFom	%DM	39.40	30.3 - 45.4
Lignin	%NDFom	16.88	
NDFD12	%NDFom	49.31	32.2 - 46.2
NDFD 24	%NDFom	53.38	30.3 - 51.0
NDFD 30	%NDFom	55.61	36.2 - 55.3
NDFD 48	%NDFom	58.88	41.4 - 60.9
NDFD240	%NDFom	61.85	44.9 - 66.6
uNDFom12	%DM	19.97	18.3 - 28.6
uNDFom24	%DM	18.37	15.9 - 28.6
uNDFom30	%DM	17.49	15.0 - 27.0
uNDFom48	%DM	16.20	14.3 - 25.5
uNDFom240	%DM	15.03	11.9 - 23.7
Sugar (ESC)	%DM	0.01	0.99 - 6.[illegible]0
Sugar (WSC)	%DM	1.75	1.36 - [illegible].20
Starch	%DM	3.39	0.[illegible] - 3.91
Fat (EE)	%DM	6.72	[illegible].47 - 4.43
TFA (fat)	%DM	2.07	1.08 - 3.16
16:0 Palmitic	%TFA	28.50	22.0 - 33.7
18:0 Stearic	%TFA	3.86	3.65 - 7.06
18:1 Oleic	%TFA	3.38	2.63 - 6.59
18:2 Linoleic	%TFA	22.22	20.9 - 30.6
18:3 Linolenic	%TFA	40.10	22.0 - 47.3
Ash	%DM	13.59	9.01 - 14.4
Calcium	%DM	1.59	1.02 - 1.60
Phosphorus	%DM	0.36	0.29 - 0.43
Magnesium	%DM	0.38	0.24 - 0.40
Potassium	%DM	4.38	2.13 - 3.53
Sulfur	%DM	0.32	0.19 - 0.32
Chloride	%DM	0.73	0.2[illegible] - 1.18
Lactic Acid	%DM	0.1[illegible]	0.00 - 9.74
Acetic Acid	%DM	9.02	0.00 - 6.2[illegible]
Propionic Acid	%DM	2.18	0.00 - [illegible].47
Butyric Acid	%DM	13.76	0.[illegible] - 0.95
Iso-Butyric Acid	%DM	0.81	[illegible].00 - 0.85
Ethanol	%DM	0.77	0.00 - 1.35
Methanol	%DM	0.[illegible]1	0.00 - 2.81
1 Propanol	%DM	[illegible]0.01	0.00 - [illegible].66
1,2 Propanediol	%DM	0.17	0.[illegible] - 0.57
2 Butanol	%DM	<0.01	0.00 - 0.31
Silage Acids	%DM	25.1[illegible]	

*Alfalfa haylage statistics provided for comparison.

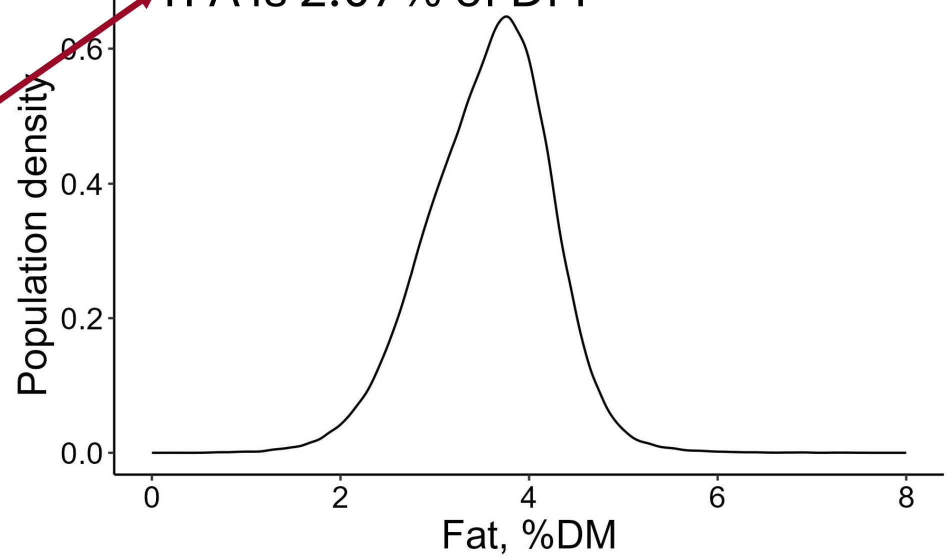
Calculations

NFC	%DM	27.20		
NSC	%DM	5.14		
RFV		138.16		
RFQ		157.39		
NDF kd rate MIR_P1	%/hr	8.12		
Adjusted Crude Protein	%DM	18.26		
		ADF	**OARDC**	**MLK 2013**
TDN		62.10	56.45	63.18
Nel 3x	Mcal/cwt	63.78	57.41	59.91
Neg	Mcal/cwt	32.92	31.86	40.84
Nem	Mcal/cwt	58.89	57.73	67.65
Milk per ton	lbs/ton			2706

Alfalfa haylage with 6.72% Fat (EE)

Butyric acid is 13.76%!

TFA is 2.07% of DM



Is it weird or is it wrong?

Moisture	69.95%		
Dry Matter	30.05%		
pH	5.47		
		Dry Basis	**90% Range***
Crude Protein	%DM	15.53	7.99 - 19.7
AD-ICP	%CP	13.08	.28 - 23.7
ND-ICP w/SS	%CP	39.34	
Protein Sol.	%CP	42.63	0.6 - 63.8
Ammonia-CP	%CP	42.50	.24 - 13.5
Total Amino Acids	%DM	6.70	.20 - 15.5
Total Amino Acids	%CP	43.14	1.2 - 96.6
Lysine	%CP	.84	.03 - 5.95
Methionine	%CP	.09	.34 - 2.65
Isoleucine	%CP	1.80	91 - 4.40
Leucine	%CP	3.28	.34 - 8.33
Histidine	%CP	0.13	.61 - .04
ADF	%DM	49.88	6.6 - 46.
aNDF	%DM	71.75	0.5 - 67.4
aNDFom	%DM	62.31	7.3 - 63.1
Lignin	%NDFom	9.24	
Lignin (Sulfuric Acid)	%DM	5.76	.26 - 9.81
NDFD12	%NDFom	60.44	1.9 - 71.0
NDFD 30	%NDFom	69.11	1.3 - 78.3
NDFD 120	%NDFom	78.99	5.6 - 89.7
NDFD240	%NDFom	82.91	4.5 - 89.9
uNDFom12	%DM	24.65	2.4 - 40.8
uNDFom30	%DM	19.25	.24 - 34.3
uNDFom120	%DM	13.09	.01 - 25.8
uNDFom240	%DM	10.65	.29 - 26.2
Sugar (ESC)	%DM	0.20	08 - 7.93

18:3 Linolenic	%TFA	39.47	29.1 - 58.6
Ash	%DM	16.31	5.71 - 12.6
Calcium	%DM	0.43	0.22 - 1.09
Phosphorus	%DM	0.33	0.24 - 0.43
Magnesium	%DM	0.33	0.15 - 0.34
Potassium	%DM	1.78	1.17 - 3.56
Sulfur	%DM	0.16	0.13 - 0.30
Chloride	%DM	0.10	0.19 - 1.44
Lactic Acid	%DM	1.74	0.38 - 8.11
Acetic Acid	%DM	3.30	0.13 - 2.90
Propionic Acid	%DM	0.70	0.07 - 0.56
Butyric Acid	%DM	2.81	0.03 - 1.53

*Mixed grass silage statistics provided for comparison.

Calculations

NFC	%DM	9.82			
NSC	%DM	4.22			
RFV		64.78			
RFQ		92.06			
DF kd rate MIR_P1	%/hr	6.56			
Adjusted Crude Protein	%DM	14.77			
			ADF	**OARDC**	**MLK 2013**
TDN	%DM		50.04	47.93	52.65
Nel 3x	Mcal/cwt		50.34	47.92	54.67
Neg	Mcal/cwt		25.07	18.77	25.90
Nem	Mcal/cwt		50.34	43.57	51.23
Milk per ton	lbs/ton				2396

Butyric acid = 2.81%DM

Ash = 16.31%DM

Ammonia-CP = 42.5% of CP

Lysine = 0.84%CP

Crude protein = 15.53%DM

Total amino acids = 6.70%DM, 43.14%CP

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Leucine	%CP	3.28	.34 - 8.33
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Lignin (Sulfuric Acid)	%DM	5.76	.26 - 9.81
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NDFD 30	%NDFom	69.11	1.3 - 78.3
NDFD 120	%NDFom	78.99	5.6 - 89.7
NDFD240	%NDFom	82.91	4.5 - 89.9
uNDFom12	%DM	24.65	2.4 - 40.8
uNDFom30	%DM	19.25	.24 - 34.3
uNDFom120	%DM	13.09	.01 - 25.8
uNDFom240	%DM	10.65	.29 - 26.2
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Calcium	%DM	0.43	0.22 - 1.09
Phosphorus	%DM	0.33	0.24 - 0.43
Magnesium	%DM	0.33	0.15 - 0.34
Potassium	%DM	1.78	1.17 - 3.56
Sulfur	%DM	0.16	0.13 - 0.30
Chloride	%DM	0.10	0.19 - 1.44
Lactic Acid	%DM	1.74	0.38 - 8.11
Acetic Acid	%DM	3.30	0.13 - 2.90
Propionic Acid	%DM	0.70	0.07 - 0.56
Butyric Acid	%DM	2.81	0.03 - 1.53

*Mixed grass silage statistics provided for comparison.

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RFV		64.78		
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NDF kd rate MIR_P1	%/hr	6.56		
Adjusted Crude Protein	%DM	14.77		
		ADF	**OARDC**	**MLK 2013**
TDN	%DM	50.04	47.93	52.65
Nel 3x	Mcal/cwt	50.34	47.92	54.67
Neg	Mcal/cwt	25.07	18.77	25.90
Nem	Mcal/cwt	50.34	43.57	51.23
Milk per ton	lbs/ton			2396

Lysine = 0.84%CP

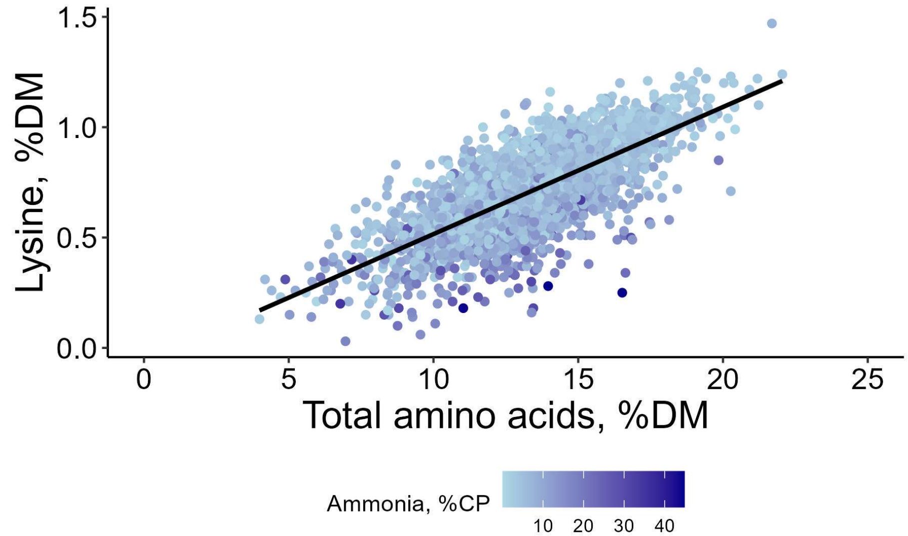
Crude protein = 15.53%DM

Total amino acids = 6.70%DM, 43.14%CP

Butyric acid = 2.81%DM

Ash = 16.31%DM

Ammonia-CP = 42.5% of CP



Is it weird or is it wrong?

Moisture	10.23%
Dry Matter	89.77%

		Dry Basis	90% Range*
Crude Protein	%DM	106.49	87.5 - 100.0
Total Amino Acids	%DM	92.17	79.4 - 99.0
Total Amino Acids	%CP	86.55	88.3 - 100.0
Lysine	%CP	7.65	5.85 - 8.84
Methionine	%CP	0.56	0.65 - 1.33
Cysteine	%CP	0.53	
Alanine	%CP	6.94	
Aspartic Acid	%CP	10.26	
Glutamic Acid	%CP	7.20	
Glycine	%CP	4.18	
Isoleucine	%CP	0.31	0.29 - 2.60
Leucine	%CP	11.32	9.49 - 12.8
Proline	%CP	2.62	
Threonine	%CP	2.65	
Valine	%CP	7.40	
Arginine	%CP	3.45	
Histidine	%CP	6.52	4.36 - 6.96
Phenylalanine	%CP	6.71	
Tyrosine	%CP	2.75	
Tryptophan	%CP	1.86	
Serine	%CP	3.64	

*Blood meal statistics provided for comparison.

Blood meal with crude protein > 100%DM

Total amino acids are 92.17%DM

Is it weird or is it wrong?

Moisture	6.61%		
Dry Matter	93.39%		
		Dry Basis	**90% Range***
Crude Protein	%DM	14.13	7.06 - 16.9
aNDF	%DM	2.61	0.93 - 20.4
aNDFom	%DM	2.29	0.63 - 19.5
Sugar (WSC)	%DM	21.98	11.9 - 46.3
Starch	%DM	55.82	29.7 - 66.2
Fat (EE)	%DM	7.63	2.26 - 15.1
Ash	%DM	4.32	1.89 - 7.17

*Bakery waste statistics provided for comparison.

WATER SOLUBLE STARCH	%DM	22.93	
Sugar (ESC)	%DM	10.78	8.71 - 37.7
Total sugars (HPIC)	%DM	6.51	

Mass balance = 106.17 %DM

	% of sugar
Glucose	21.94
Arabinose	0.00
Xylose	0.00
Galactose	0.44
Fructose	19.57
Lactose	4.17
Sucrose	0.60
Raffinose	0.00
Stachyose	0.00
Maltose	53.02

Conclusions

- Anomalies in feed analysis challenge database development
- Statistical filtering often removes outlier samples
  (Yoder et al., 2014; Tran et al., 2020)
- True biological variation may be mistaken for analytical error
- Opportunity: Rethink database curation strategies
  - Distinguish between analytical error and true anomalies caused by real variation

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Thank you!

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