

# How to get the most out of your NIR instrument

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# Agenda

- What is NIR and what can it be used for?
- How good can an NIR be
- Validation and adjustment
- Summary

**What can NIR measure and how accurate  
can it be?**

# Color = concentration



# We need a calibration

The NIR instrument needs to be trained to recognize different concentrations

1. Analyze a set of samples with reference method and NIR
2. The data is combined in the calibration software

An equation describing the relationship between the absorbance at specific wavelengths and the composition of the sample

# What NIR can and cannot do

- Can measure
  - Moisture
  - Organic constituents like protein, starch, fat
  - Ash
- Cannot measure
  - Very low concentrations
  - Things which don't absorb infrared light
- Can sometimes measure
  - Functional properties: Water absorption, Starch damage

# NIR instrument techniques

Used for about 30 years in agriculture

- Filter Instruments – 35 years old
- Scanning monochromators – 35 years old
- Fourier Transform (FT) – 25 years old
- Diode Array – available since 20 years

All three advanced techniques are very good

Choose a supplier you trust

# How good can NIR be?

The NIR instrument is trained using a reference method and inherits the accuracy of that method

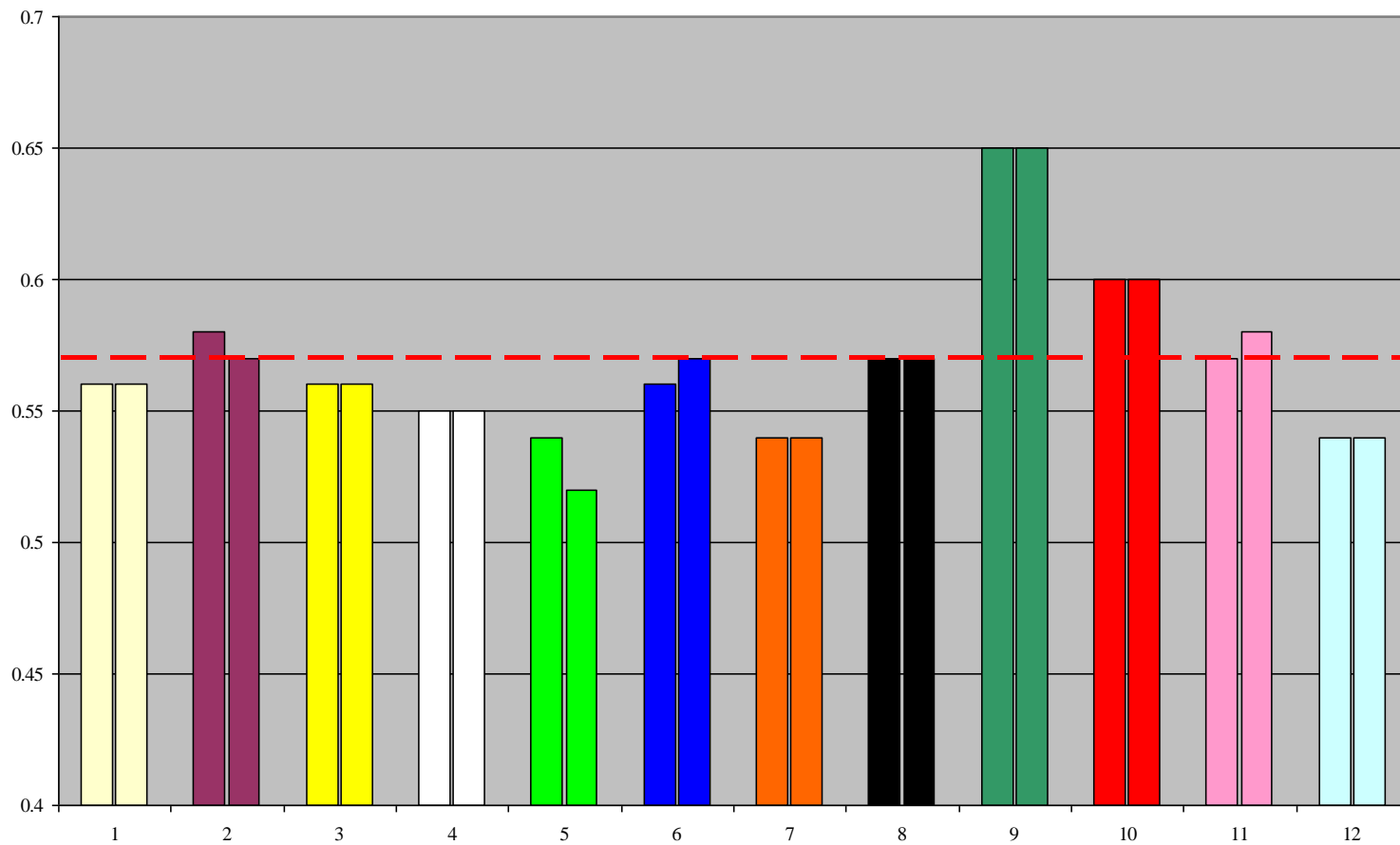


1-1 ½





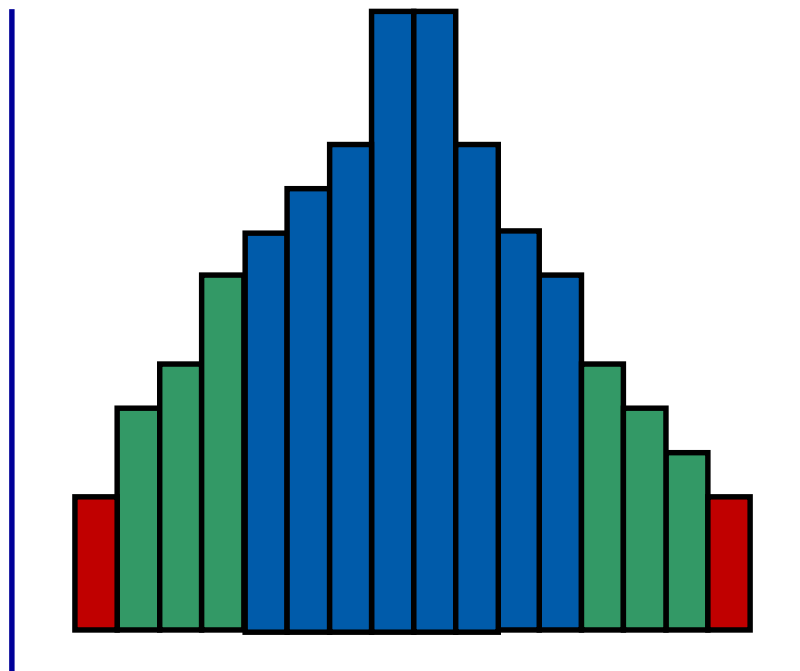
# Is the reference method always right?



# Typical reference method accuracies

Moisture	0.1-0.3
Protein	0.1-0.3
Ash	0.02-0.04

Standard Deviation (SD)

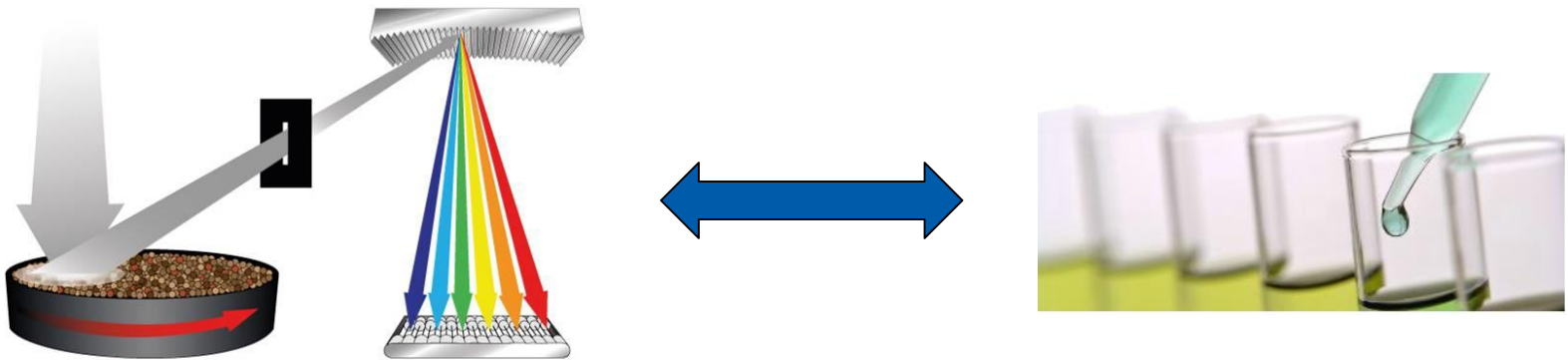


- 68 %  $\leq$  1 SD
- 95 %  $\leq$  2 SD
- 99.7 %  $\leq$  3 SD

# Validating an NIR calibration

# The purpose of validation

NIR is a secondary / indirect technique



1. Is the calibration suitable for my situation?
2. Do I need to adjust it to match my lab?

# When is validation necessary?

- At installation of new calibration
- On a regular basis to verify performance  
Analyze a few samples per week and evaluate when you have enough measurements
- If you make major changes  
New wheat supply, new production equipment, new lab equipment



# Follow guidelines and standards



ISO 12099:2010

- At least 20 samples



ICC 202

- At least 20 samples



ASTM E1655-97 NIR Std

- At least 20 samples
- 4 x no factors (dimensions) used in calibration

# Evaluate results

Compare NIR values with reference values

Example of protein in wheat

ID	LAB	NIR	LAB-NIR Diff	ID	LAB	NIR	LAB-NIR Diff
1	10.6	12	-1.4	11	14.1	14.7	-0.6
2	11.5	12.2	-0.7	12	14	15	-1
3	11.3	12.4	-1.1	13	14.2	15.2	-1
4	12	12.8	-0.8	14	14.3	15.5	-1.2
5	11.8	13	-1.2	15	14.9	15.7	-0.8
6	12.3	13.3	-1	16	14.8	15.9	-1.1
7	12.1	13.4	-1.3	17	15.5	16.2	-0.7
8	13.2	13.8	-0.6	18	15.1	16.5	-1.4
9	13.1	14	-0.9	19	15.7	16.6	-0.9
10	13.4	14.3	-0.9	20	16.2	17	-0.8

On average NIR is too high by 1.0

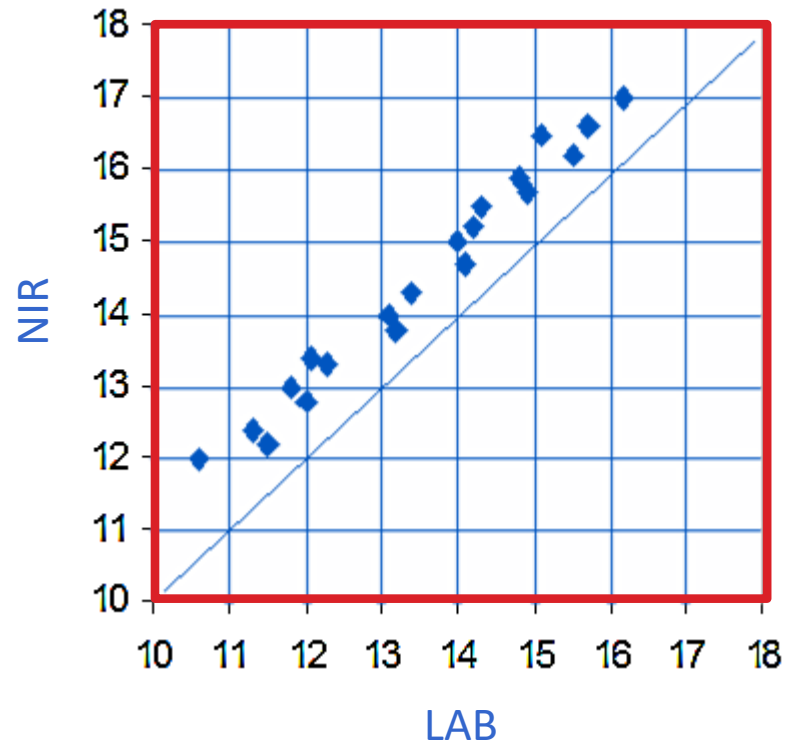
Standard Deviation NIR-Lab is 0.24

# Adjusting a calibration

**Bias is average difference, LAB vs NIR**

EXAMPLE Bias = -1

- NIR gives too high results
- Subtract 1 from NIR predictions





# Validation set composition

- Needs to cover expected variation in routine analysis
- Full variation in major constituents
- Other important variation such as:
  - Geographical origin
  - Annual variation
  - Varieties
  - Changes in production
  - Sub products
  - Particle size
  - Temperature



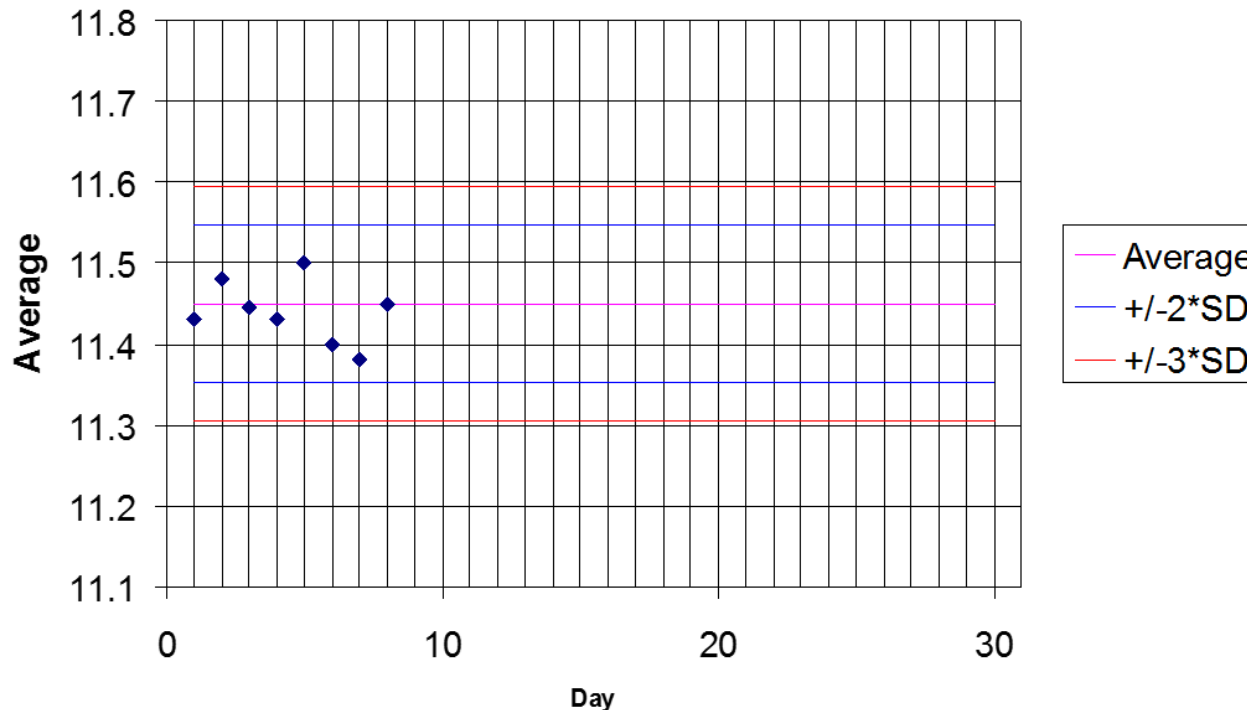
Don't start creating different groups with different adjustments!

# What if results don't look good?

- Keep in mind that your reference method isn't perfectly accurate
- If Standard Deviation between NIR and Lab is  $>1.5$  times the accuracy of your reference method you should look into it
- Unless you have lots of experience, ask your NIR supplier for assistance
- Usual reasons
  - Reference method errors
  - Calibration isn't suitable
  - Handling
  - Instrument issues

# Check Sample

- Analyze a check sample on a daily basis
- The sample must be stable and not change
- Artificial material is preferred
- Plot results and evaluate



# Bonus material

# Samples change

- Minimize time between NIR and LAB analyses
  - Moisture loss, degradation, moulds, insects
- Accredited lab (moisture air oven)
  - Moisture loss in samples during one month
  - Samples were stored in sealed plastic bags



Product	Moisture 1 (%)	Moisture 2 (%)	Diff (%)
Corn	13.5	10.4	3.1
Canola	6.8	5.2	1.6
Rice	14.6	11.2	3.4

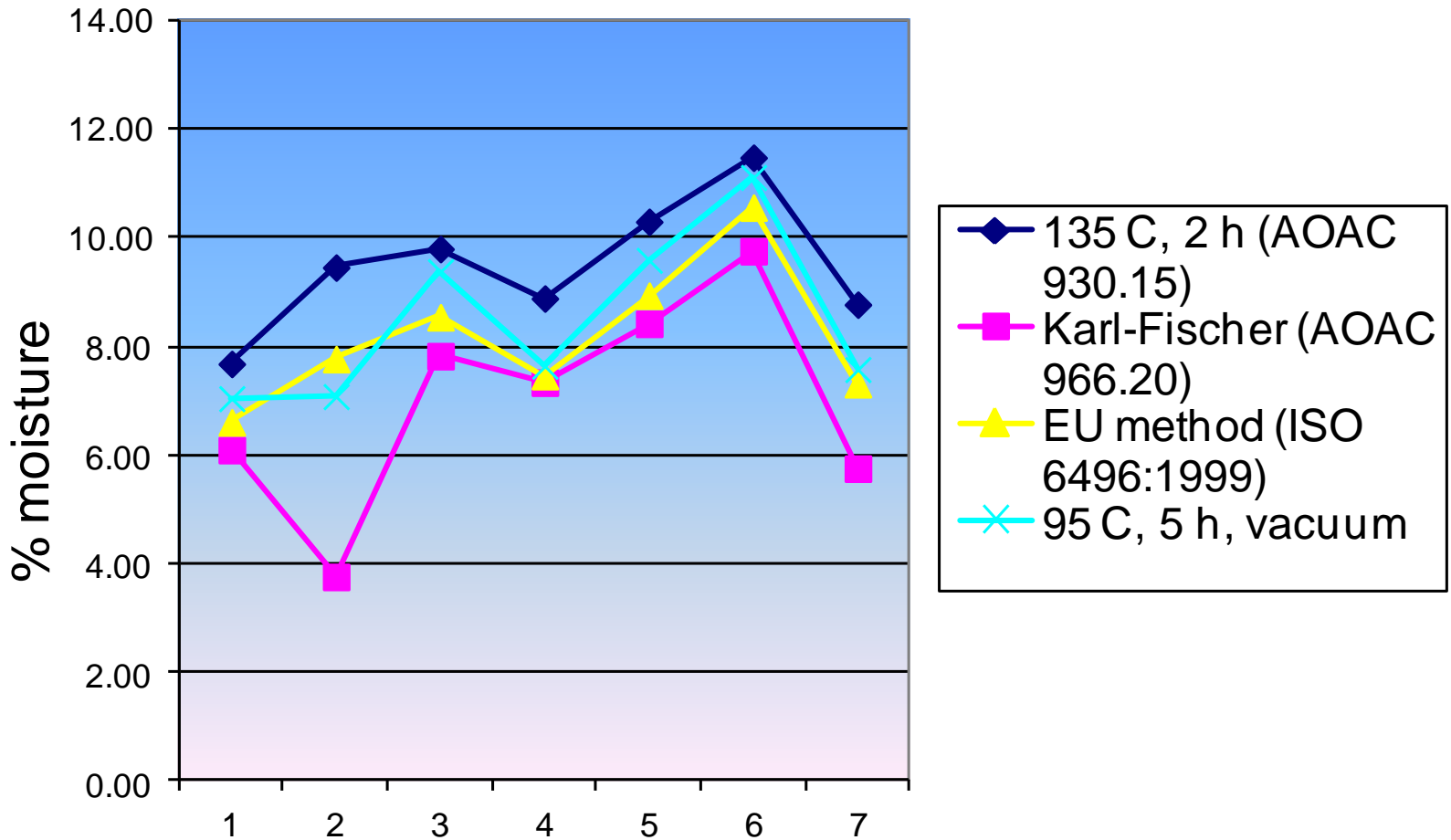
# Sample storage

- In refrigerator
- Vacuum pack
- Air tight sample containers
  - Screw cap or tight lid
  - Metal, glass or plastic
  - Completely filled with sample



# Reference methods are different

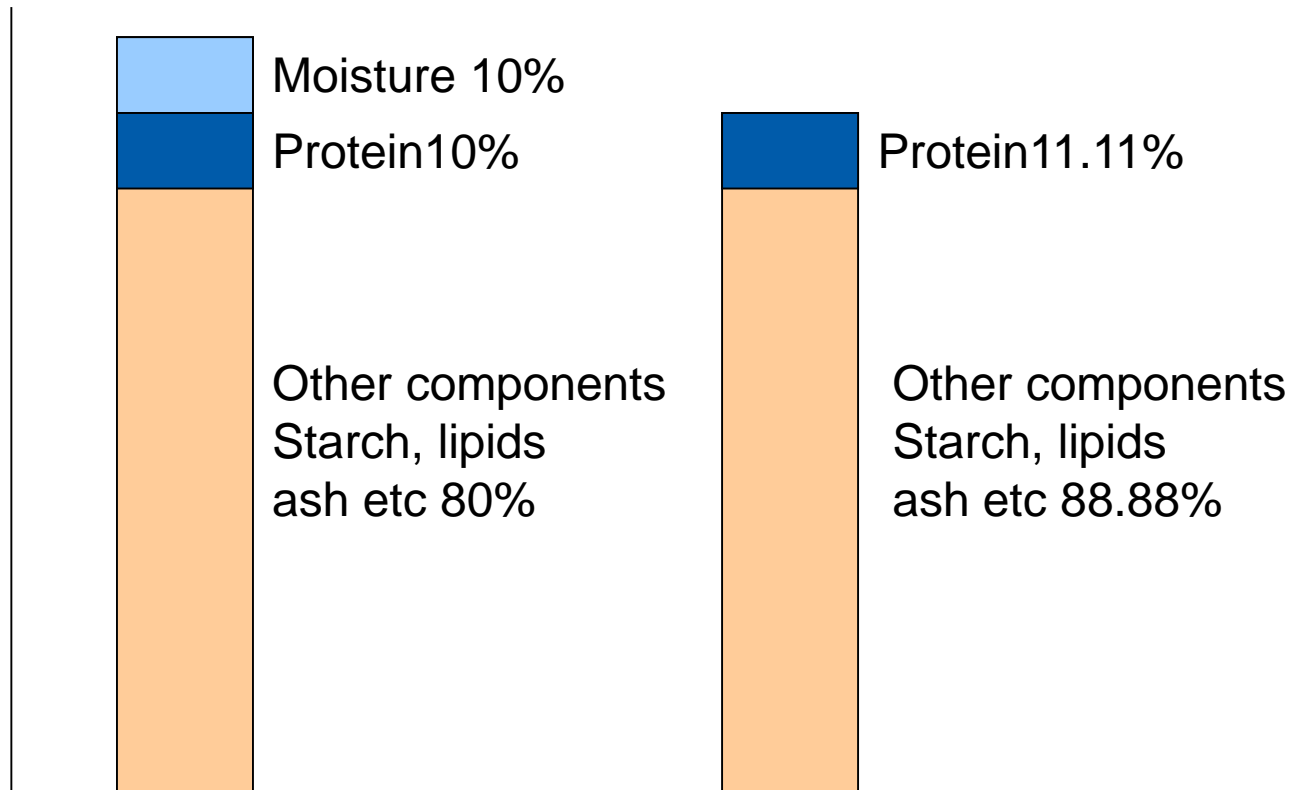
Moisture by different methods



# Moisture basis

**Protein 10/100 = 10%  
of total sample  
- As is basis**

**Protein 10/90 = 11.11%  
of total dry material  
- The dry basis**





# Sampling error

- Sampling error has substantial impact on performance
- Analyze *same sample* with NIR and LAB



# Summary

- NIR is excellent for moisture, major organic constituents and ash. Can approximate a few other things.
- Several instrument technologies can be suitable
- Make sure you have a calibration which covers what you want to measure, and validate it
- Understand your reference method error and accept it
- Use daily check sample and regular comparison against reference method
- Always follow guidelines and standards when making adjustments
- Ask an expert

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