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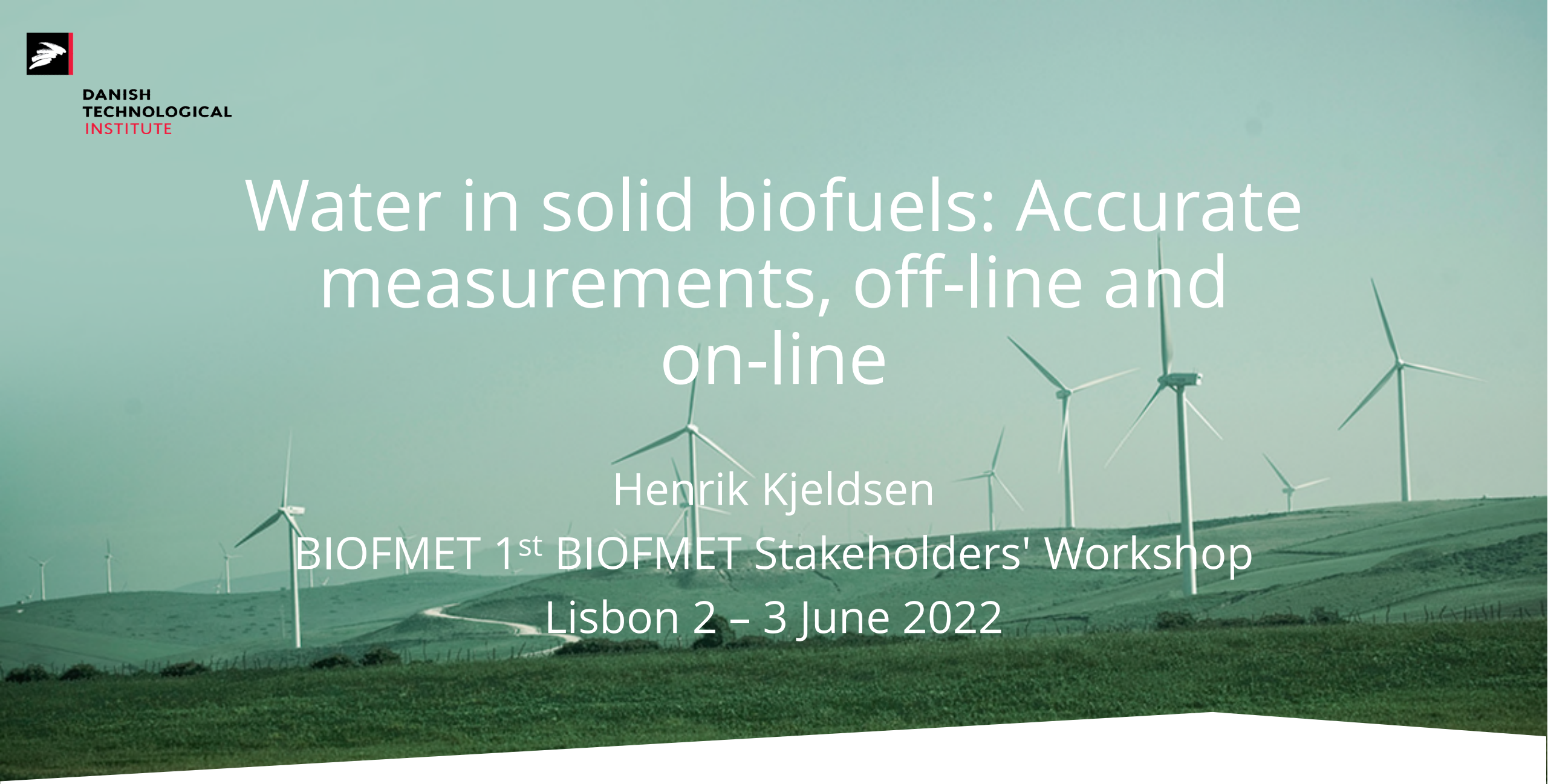
# Water in solid biofuels: Accurate measurements, off-line and on-line

Henrik Kjeldsen

BIOFMET 1st BIOFMET Stakeholders' Workshop
Lisbon 2 – 3 June 2022



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# *How much water?*

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# Teaser / outline: Water in solid biofuels

- Solid biofuel = Combustible organic compounds + lot of water (20 – 50 %)
- Organic compounds burn – water don’t

1. How to measure water content
2. Traceability
   - Reference method
   - Transfer to industry

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# Motivation

- Impact
  - €€€€€€€€
  - Combustion technique
  - Sampling
- Challenges
  - Heterogeneity with respect to…
    - Sample material
    - Impurities
    - Physical parameters
    - Water content

- CPH burning solid biofuel
  - Green: $CO_2$ neutral
  - Backup for solar and wind power

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# What are the options?

- Relevant for all part of the value chain:
  - Producers
  - Distributers
  - Consumers

Options

1. LoD (Loss on Drying) + Sampling
2. Moisture measurement device (+ less sampling)

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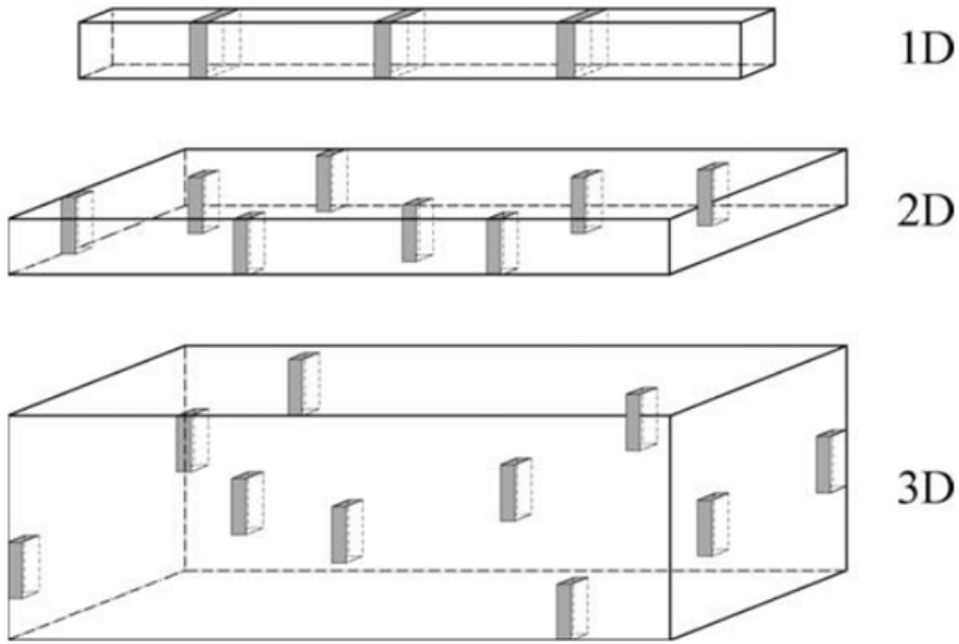
# Uncertainty / accuracy I




- Where does the uncertainty come from?
  - Sampling: 80 %
  - Handling: 15 %
  - Measurement (LoD): 5 %
  - Source: 1) Finish VVT report, 2) Similar results by DTI
- Sampling dimensions: 3D → 2D → 1D → 0D
  - Aim for 1D (*e.g. belt*) or 0D (*entire lot*)
- Classic question: Is it possible to get similar accuracy using electronic device instead of LoD for moisture measurements?
- Sampling ←→ Calibration

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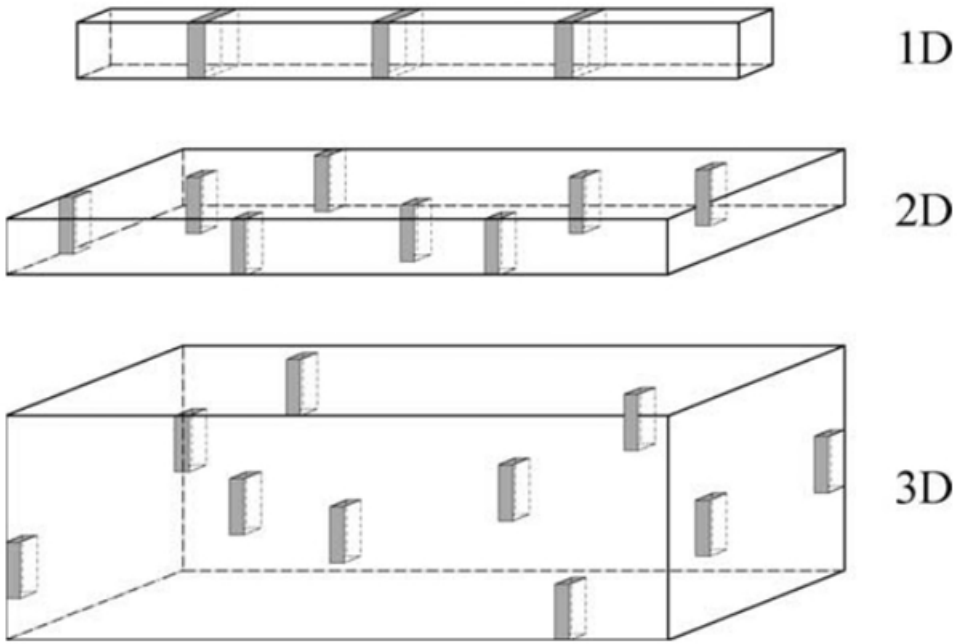
# Option 1: “Traditional” procedure




- LoD (Loss on Drying) + Sampling

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# Option 2: Online moisture measuring

- Moisture measurement device (+ less sampling)

- Where does the uncertainty come from?
  - Sampling: small
  - Handling: small
  - Measurement (device): larger
  - Calibration (larger)

- Key question: Is it possible to get similar accuracy using electronic device instead of LoD for moisture measurements?
  - Sampling ←→ Calibration

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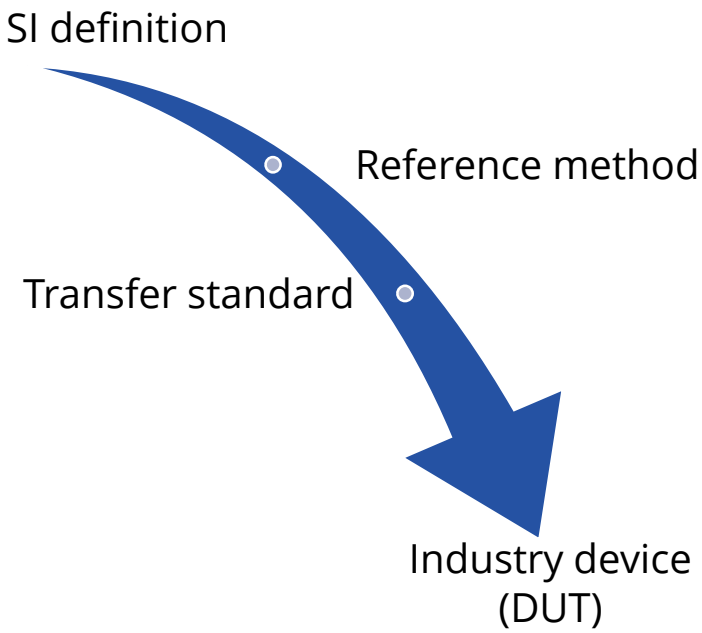
# Traceability ➔ accurate measurements in industry




- Definition of metrological traceability:
  - Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

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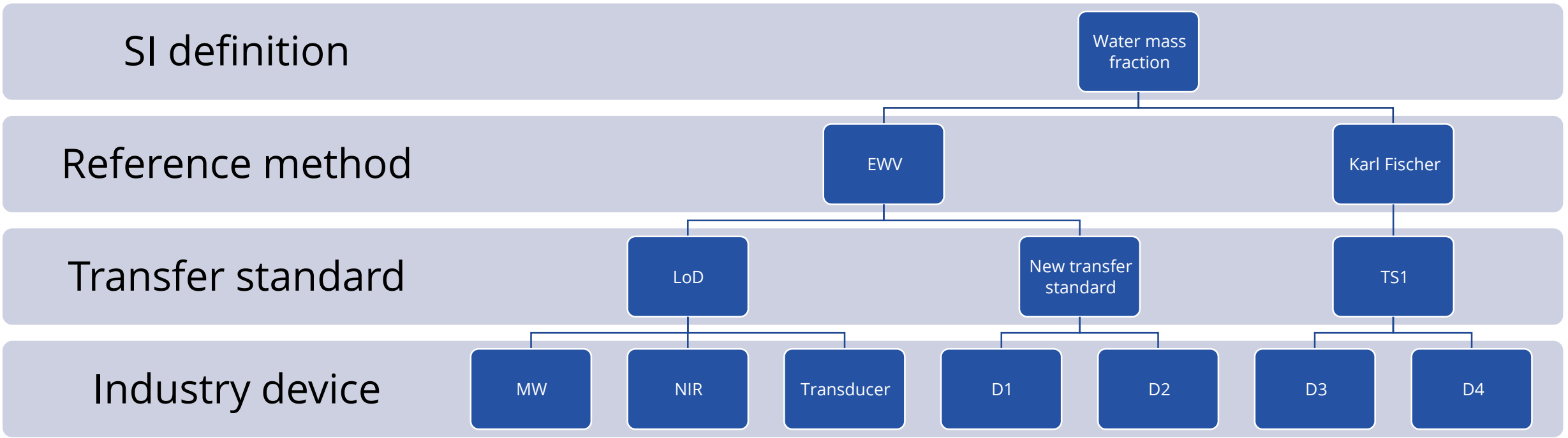
# Traceability ➔ accurate measurements in industry



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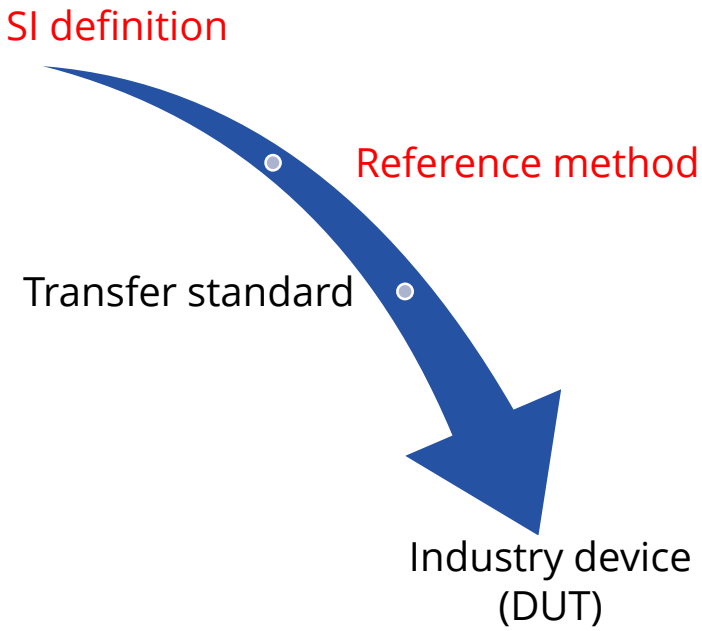
# Traceability ➔ accurate measurements in industry

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# Requirements to reference methods

- SI definition
  - Water mass fraction, $WMF$

$$WMF = \frac{m_{water}}{m_{sample}}$$

- Mass of sample, $m_{sample}$: Easy
- Mass of water, $m_{water}$: Require measuring method specific to water

- Example I: Coulometric Karl Fischer titration (cKF)
- Example II: Evolved Water Vapour (EWV)

Loss on Drying not specific to water → **NOT** a reference method

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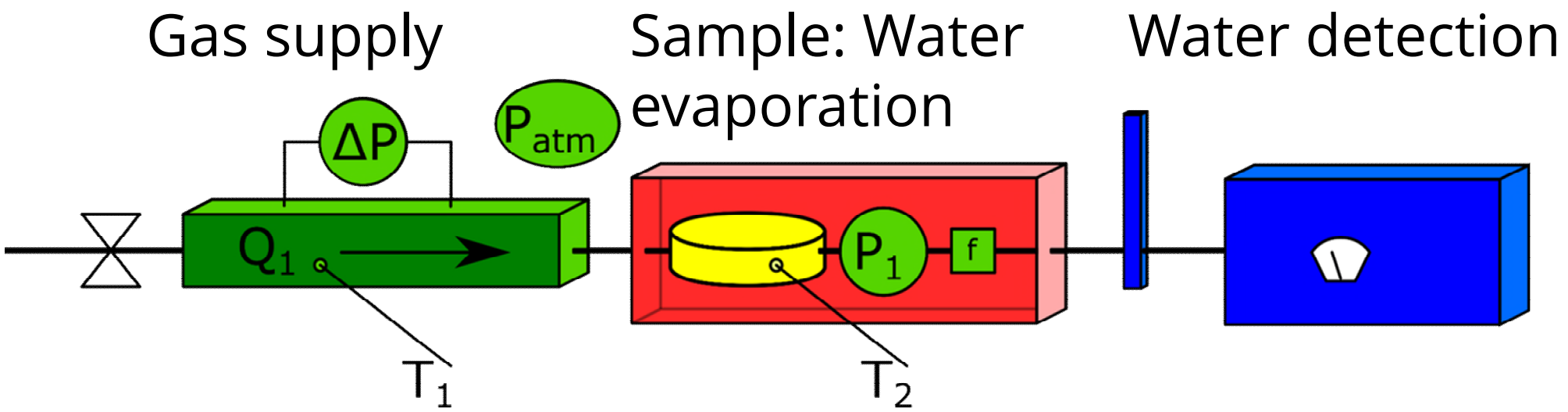
# Reference method: Evolved Water Vapour (EWV)

- Measure water vapor evolved from sample



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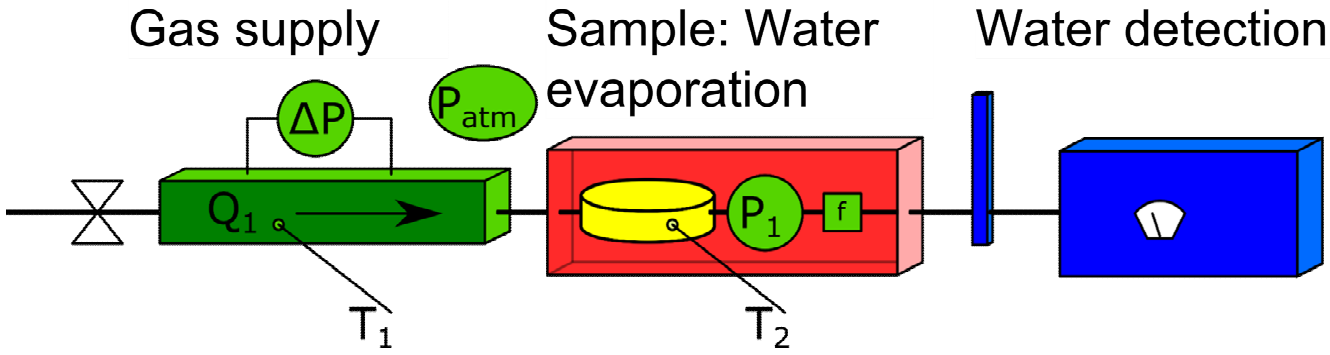


# EWV: Implementation

*Evolved Water Vapour*

- **EVW using dew-point temperature (EVW-DP) (DTI)**
  - Water = $\int [flow][water\ content]dt$
  - Measure water vapor evolved from sample
- EVW using coulometric Karl Fischer titration (vap-C-KFT) (CETIAT)
- **EVW using $P_2O_5$ sensor (CETIAT)**
- EVW-freeze (VTT)
  - Water collected by freezing

- *BIOFMET: Intercomparison ongoing*

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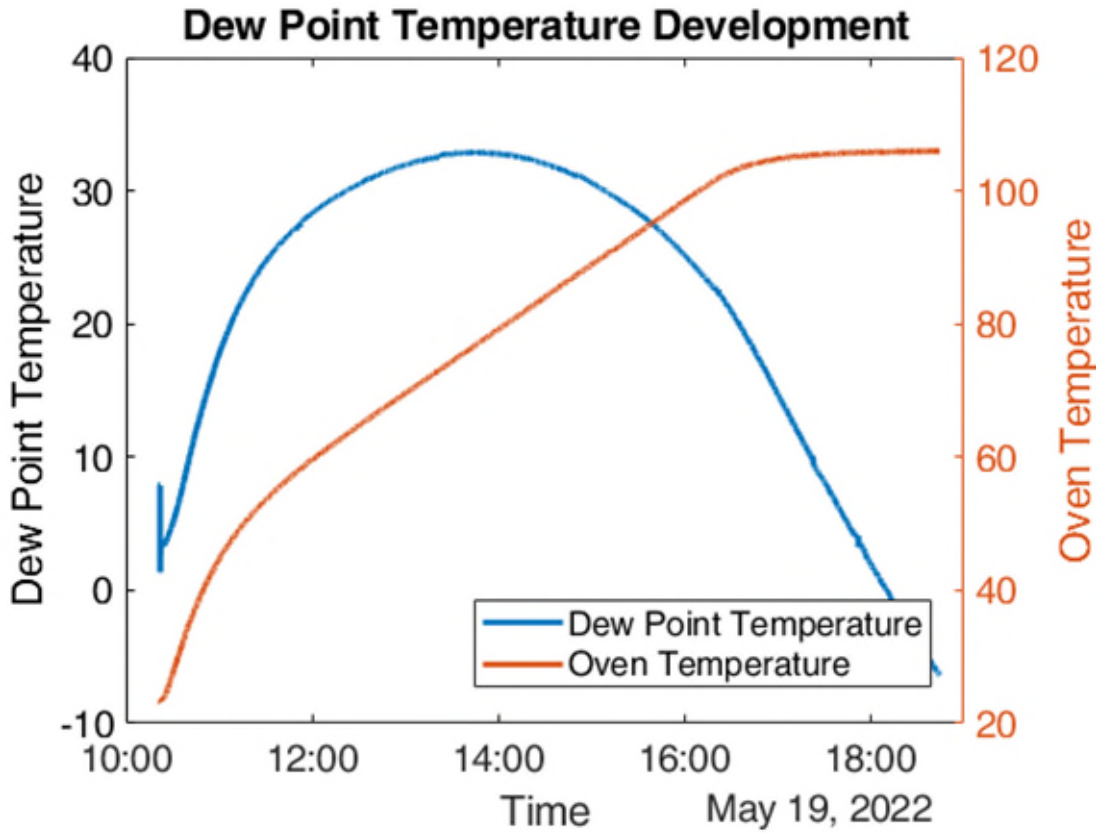
# Data from EWV-DP

- Water vapour is monitored
- Temperature is gradually increased to 105 °C
- Test ends when measured dew point is below -10 °C, corresponding to 0.1 g/h



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# EWV-DP

- Data from wood pellets
- To Be Verified: Data indicates that small, but significant amounts of VOCs are evaporated during drying

| | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 |
|---|---|---|---|---|---|
| LoD | 12.48 ± 0.09 g | 12.49 ± 0.02 g | 12.48 ± 0.01 g | 12.62 ± 0.01 g | 12.48 ± 0.01 g |
| EWV | 12.11 ± 0.30 g | 12.32 ± 0.55 g | 12.16 ± 0.38 g | 12.23 ± 0.36 g | 12.07 ± 0.34 g |
| %mc | 6.23 ± 0.02 % | 6.22 ± 0.02 % | 6.23 ± 0.02 % | 6.24 ± 0.02 % | 6.23 ± 0.02 % |
| %w | 6.05 ± 0.15 % | 6.13 ± 0.27 % | 6.07 ± 0.19 % | 6.05 ± 0.18 % | 6.03 ± 0.17 % |

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# BIOFMET intercomparison

- Results if available…

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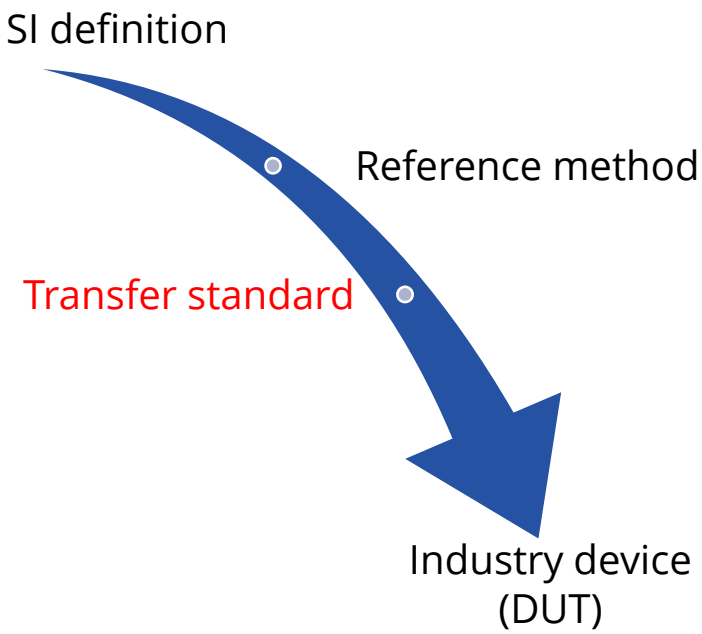
# Moving traceability to industry

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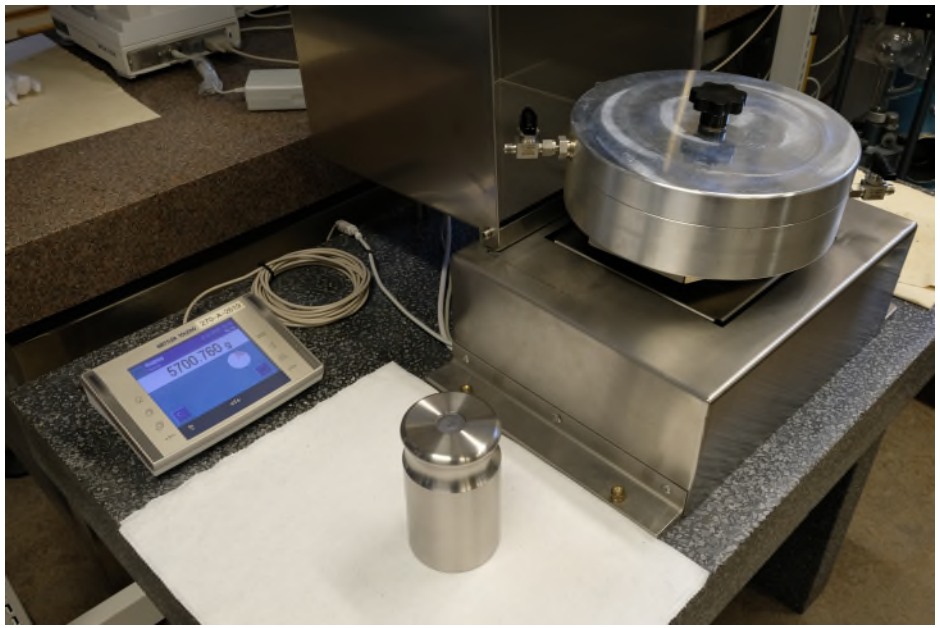
# Transfer standard

- A transfer standard is a device or method that can link an industrial device to a primary method
- Examples
  - Fresnell's device
  - Acoustic device (CMI)
  - Loss on drying
- Alternative: Certified Reference Material



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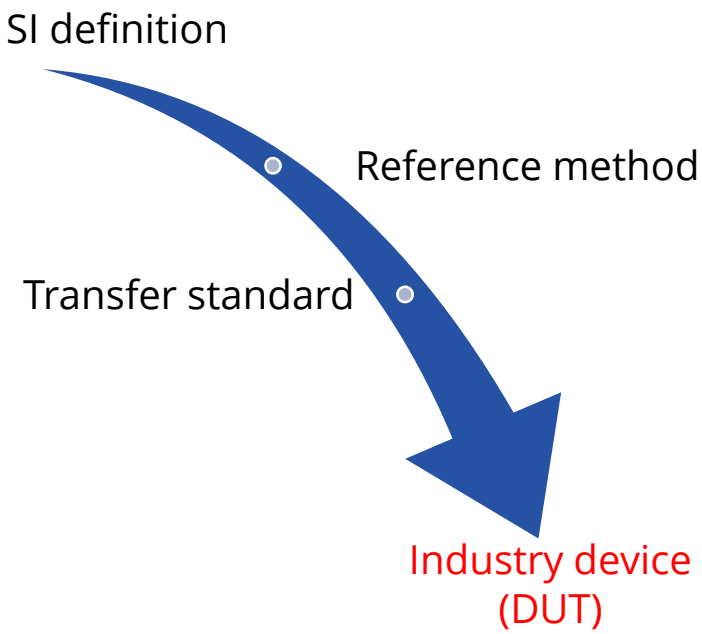
# Moving traceability to industry

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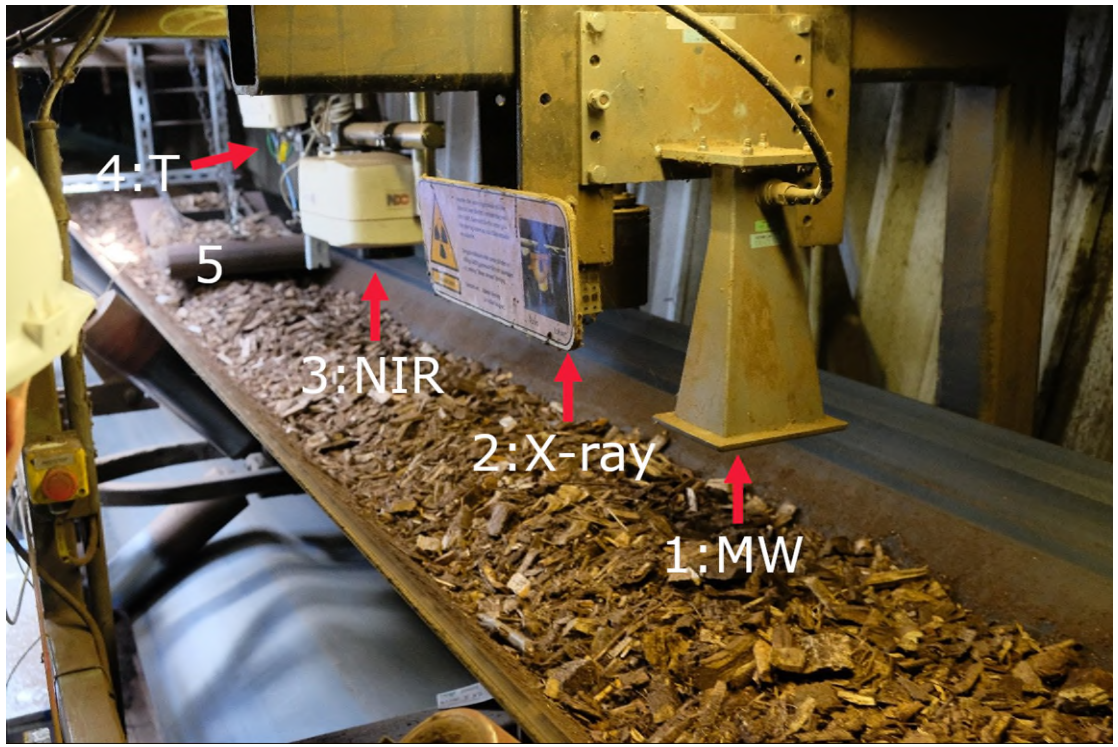
# Example of devices in industry
*DUT = Device Under Test*




- MW: MicroWave equipment
- NIR: Near InfraRed
- MT: Moisture transducer
- …

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# Calibration/adjustment: Requirements

1. Transfer standard
   - Transfer standard and DUT measurement on equivalent sample material

   →Remember this during installation!

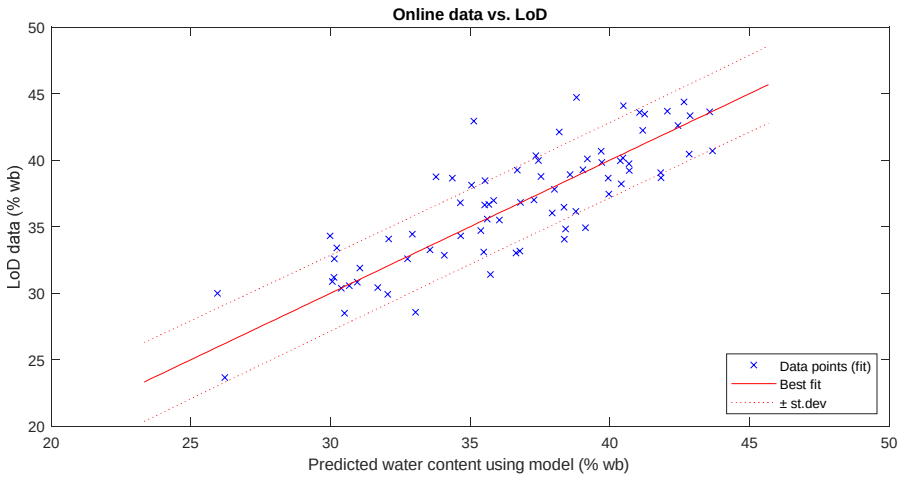
   →Sampling may be required
2. Sample material
   - Must cover entire measurement range

   →No extrapolation!
   - Special sample material may be prepared

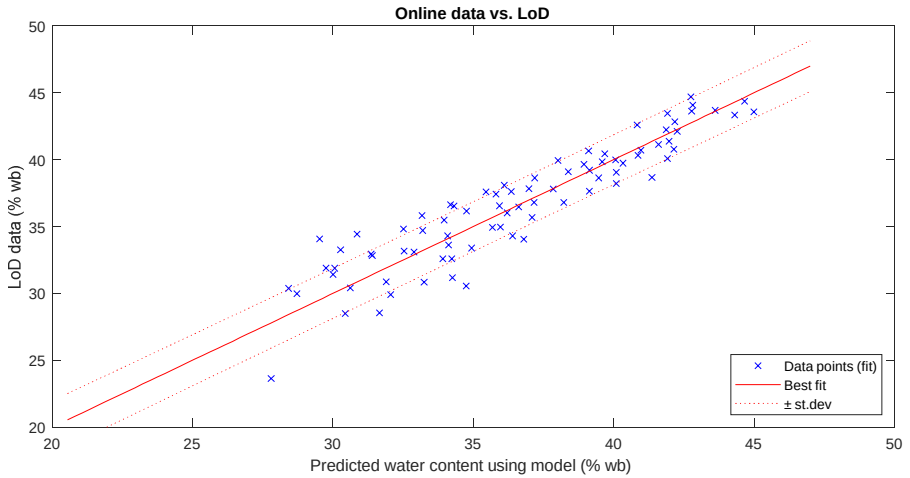
   → Control moisture content!

# Examples

## MW

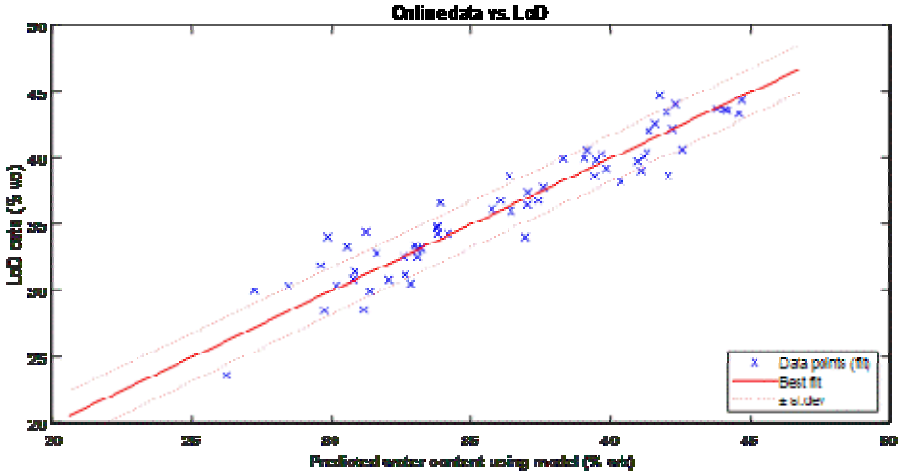



## NIR

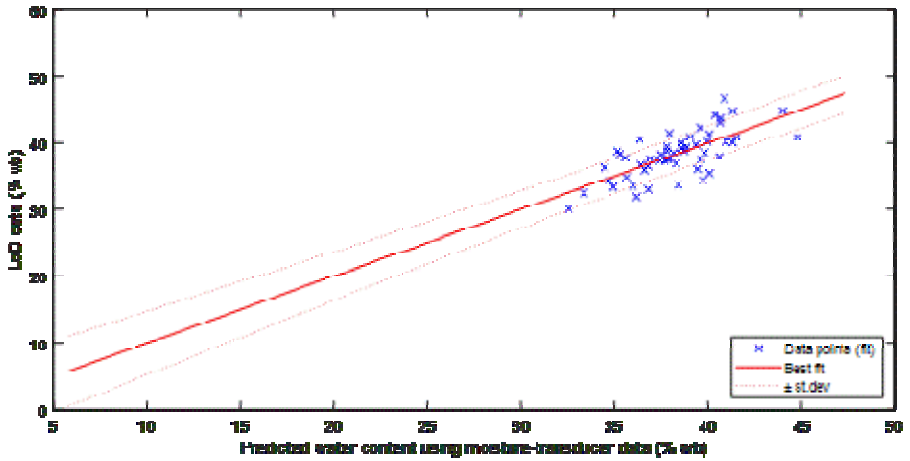


# Examples

**MW + NIR**

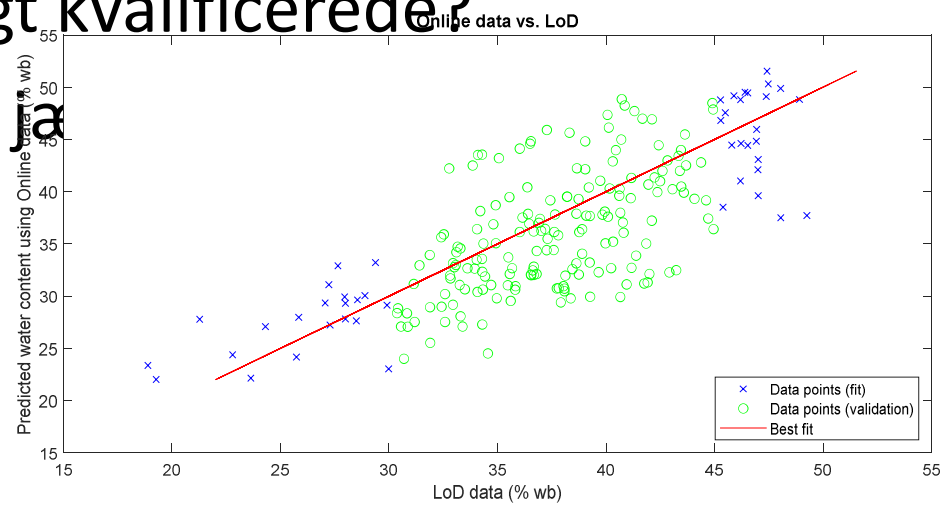



**NIR**



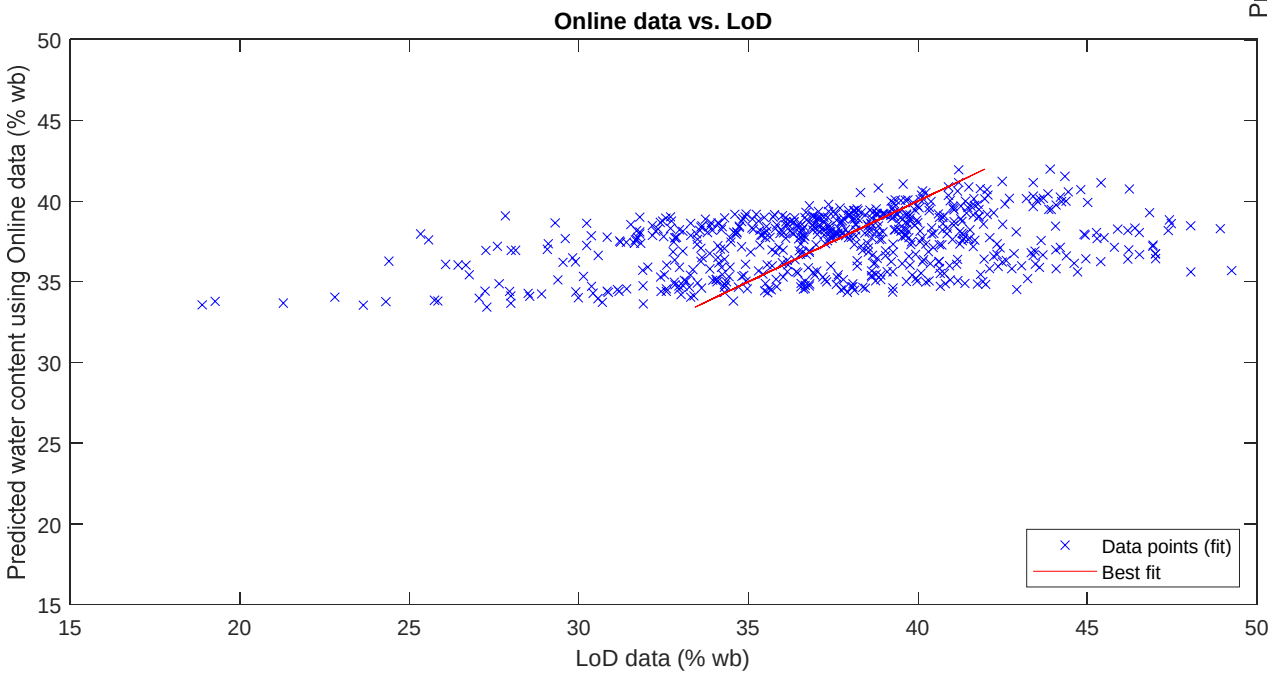
# Less successful attempts of calibration

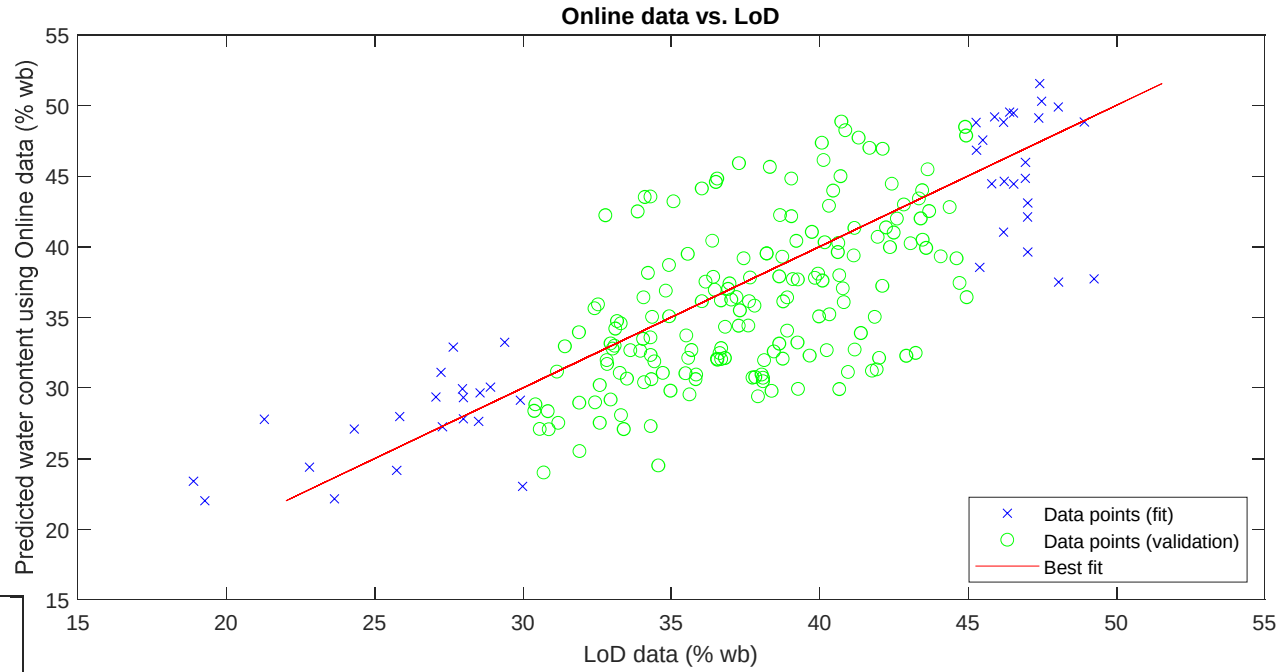
1) Måles på en repræsentativ del af flisen?
2) Er det muligt at tage prøver til kalibrering?
3) Er procedurer til måling og kalibrering fagligt kvalificerede?
4) Dækker kalibreringsprøverne måleområdet jæ



# Less successful attempts of calibration…

Data set 2




Data set 1

# Conclusion

- Two options for accurate measurement of water content
  - Sampling + offline measurements
  - Online moisture measureing → Calibration

- Calibration of online devices are possible, but requires effort

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

# Thank you for your attention!

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