DANISH TECHNOLOGICAL INSTITUTE

SoMMet

SI-traceable calibration facilities for point-scale soil-moisture sensors

Jan Nielsen, Emil Andreasen Klahn, Henrik Kjeldsen

Danish Technological Institute (DTI)

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

The Plan

1. What is SI-traceability
2. Definitions and considerations about the measurand
3. A primary measurement standard for water content in solid materials
4. A portable Loss on drying instrument for on-site calibration
5. Conclusions

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

What is traceability in metrology?

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

Metrological Traceability

- Metrological traceability is a 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

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

What is traceability in a metrological context?

- The Great Pyramid of Giza Built in the 26th century BC
  during a period of around 27 years
- Oldest and only existing of the
  “Seven Wonders of the Ancient World”

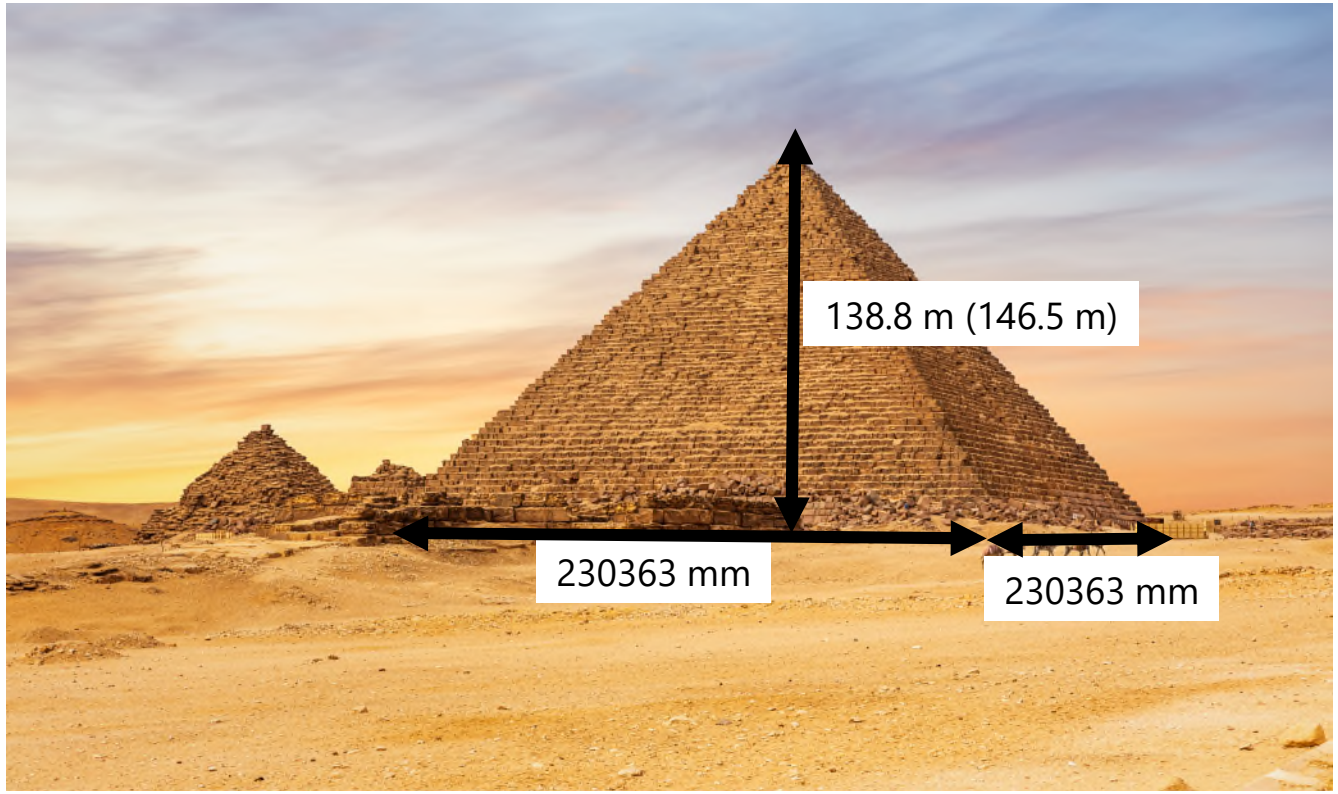
DANISH TECHNOLOGICAL INSTITUTE

SoMMet

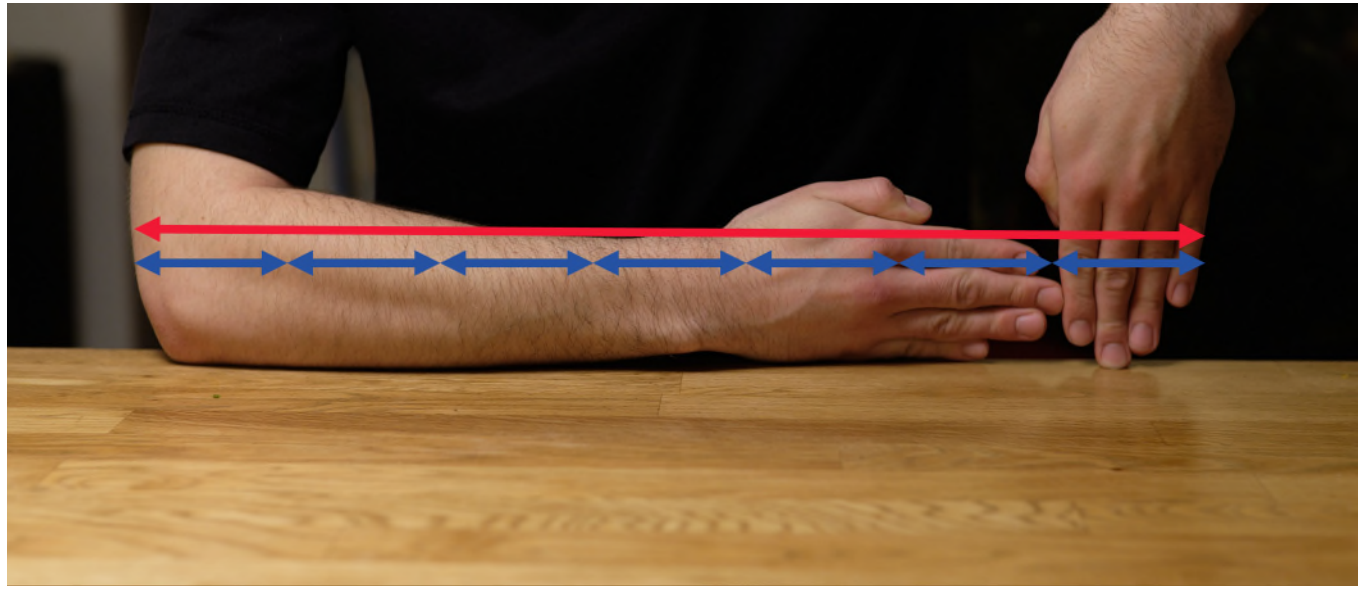
What is traceability in a metrological context?

- The Great Pyramid of Giza Built in the 26th century BC
  during a period of around 27 years
- Oldest and only existing of the
  “Seven Wonders of the Ancient World”

- The construction is an achievement in itself
- But without well-founded metrology, quality manuals and standards:
  how could it be done?



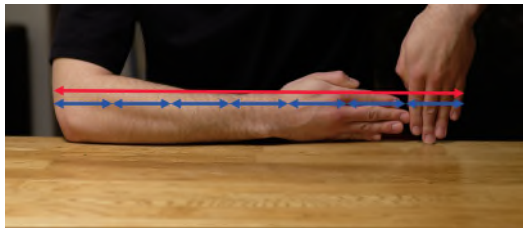
Step 1: Define a unit of length:



The cubit is based on the distance from the elbow to the middle finger of the ruling pharaoh (1 royal cubit = 523.5 to 529.2 mm)

- The royal cubit is divided into 7 palms
- A palm is divided into 4 fingers (called digit) that is: 28 digits for a cubit

Step 1: Define a unit of length:



The cubit is based on the distance from the elbow to the middle finger of the ruling pharaoh (1 royal cubit = 523.5 to 529.2 mm)

- The royal cubit is divided into 7 palms
- A palm is divided into 4 fingers (called digit) that is: 28 digits for a cubit

Step 2: Realize the unit from its definition

MUSEO EGIZIO

https://collezioni.museoegizio.it/

Step 3: Make copies – and calibrate them by comparison

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

Result:
Deviation from horizontal < 15 mm
Base length: 230363 mm ± 57 mm

Traceability, calibration and quality control

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

SI-system (2019): Definition from physical constants

- the caesium hyperfine frequency $\Delta\nu$ 9 192 631 770 Hz
- the speed of light in vacuum c 299 792 458 m/s
- the Planck constant h $6.626\,070\,15 \times 10^{-34}$ J s
- the elementary charge e $1.602\,176\,634 \times 10^{-19}$ C
- the Boltzmann constant k $1.380\,649 \times 10^{-23}$ J/K
- the Avogadro constant N_A $6.022\,140\,76 \times 10^{23}$ mol^{-1}
- the luminous efficacy of a defined visible radiation K_{cd} 683 lm/W

It is by fixing the exact numerical value of each that the unit becomes defined, since the product of the **numerical value** and the **unit** must equal the **value** of the constant.

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

The measurand – the quantity that we want to measure

See also presentation from Stephanie Bell, NPL, from MeteoMet2 MMC Soil moisture workshop 2016

- In the case of soil moisture, the measurand is the amount of water present in the soil.
- It represents the volumetric or gravimetric water content, which indicates the amount of water relative to the total volume or weight of the soil.

Straight forward?

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

To define the measurand we need to consider …

1. **Water (or moisture) content**
   - Proportion of water in sample
   - = mass of water = m_w,
   - Unit: kg
2. **Water mass fraction**
   - Proportion of water in wet matrix (i.e. wet sample)
   - = wet basis =$m_w/m_{wet\ sample}$
   - Unit: kg/kg
3. **Water mass ratio**
   - Ratio of water to dry matrix (i.e. dry sample)
   - = dry basis = $m_w/m_{dry\ sample}$,
   - Unit: kg/kg
4. **Water volume fraction**
   - Ratio of water to (wet) matrix volume
   - Requires sample density, ρ, or volume, V
   - $= m_w/V_{sample} = \frac{m_w}{m_{sample}} \cdot \rho_{sample}$
   - Unit: kg/m^3, or g/L, …
5. **…**

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

To define the measurand we need to consider …

Measurand

- Analyte = water or moisture
- Sample = soil (type, location, other specs..)
- Measured quantity = mass of water; otherwise, ratio or fraction of sample material
- Method, e.g. mass loss on drying at 105 °C

Unit

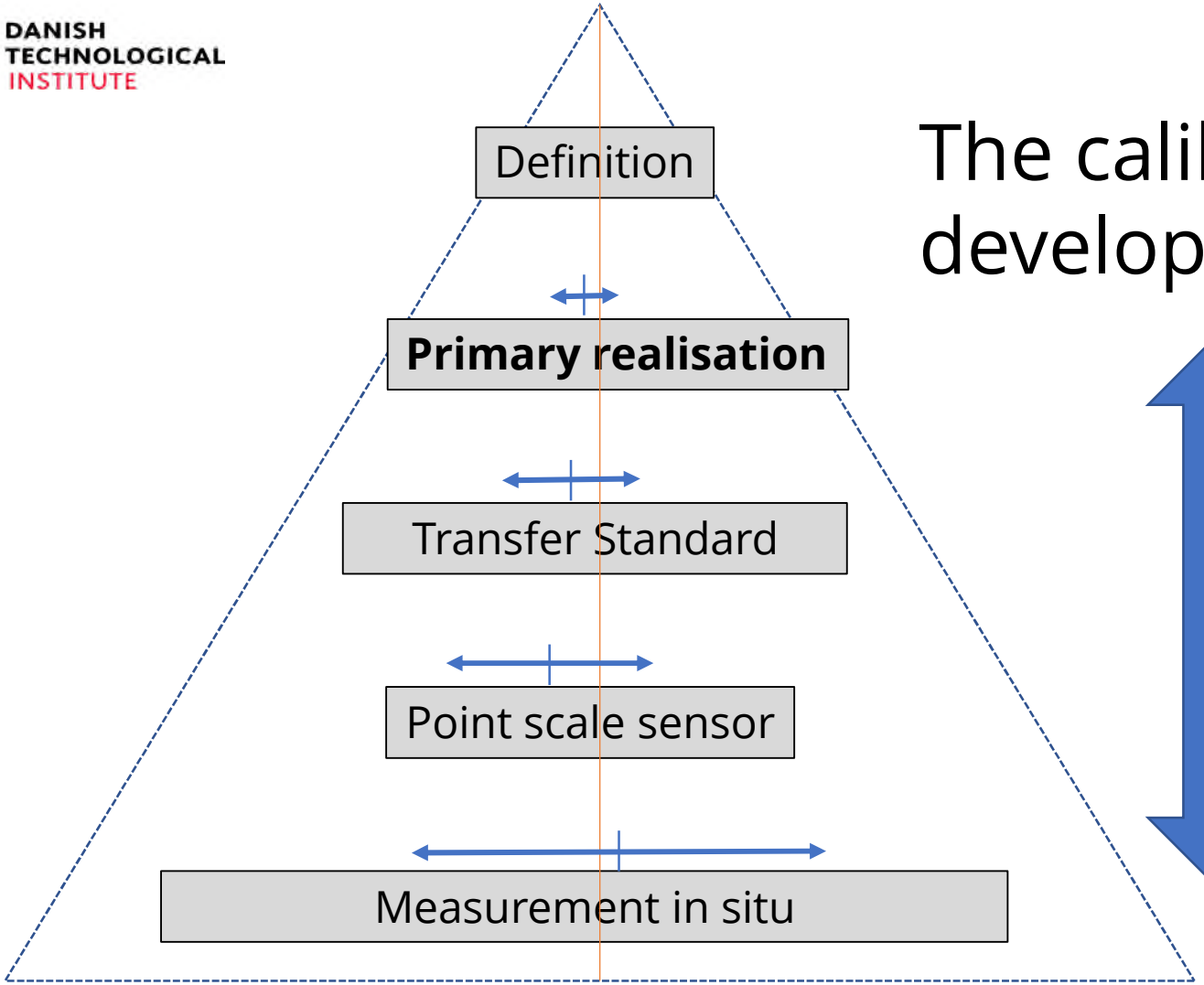
Note: Humidity / water content / moisture content

- Humidity (%rh): Relative humidity of air (*related water activity*)
- Moisture = water, volatiles, liquids
  - E.g. EN 14787:2005 (water in fertilizer) Moisture = mass loss on drying
  - **Problem 1**: Defining the dry state - Water can be present as liquid, adsorbed or chemical bound
  - **Problem 2**: Attaining a dry state (Volatiles → mass loss, Oxidation of matrix → mass increase, Loss of organic matter → mass loss
  - **Problem 3:** What is “oven dry”? (Drying time, Lab relative humidity, …)
- Water content = water
- Soil: We use “moisture” – for water content

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

The calibration hierarchy developed in SoMMet



For every step in the chain of calibrations the uncertainty increases

Too low uncertainties ”breaks” the traceability chain

Objective: *To develop metrological framework, including primary and secondary transfer standards, to ensure SI-traceable point-scale soil moisture measurements with uncertainties of 5 % under laboratory conditions.*

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

A primary measurement standard for water content in solid materials

Primary realisation

1. **Water amount**
   - Measure amount of water → Reference method
   - Making reference sample with known amount of water (→ requires dry sample)

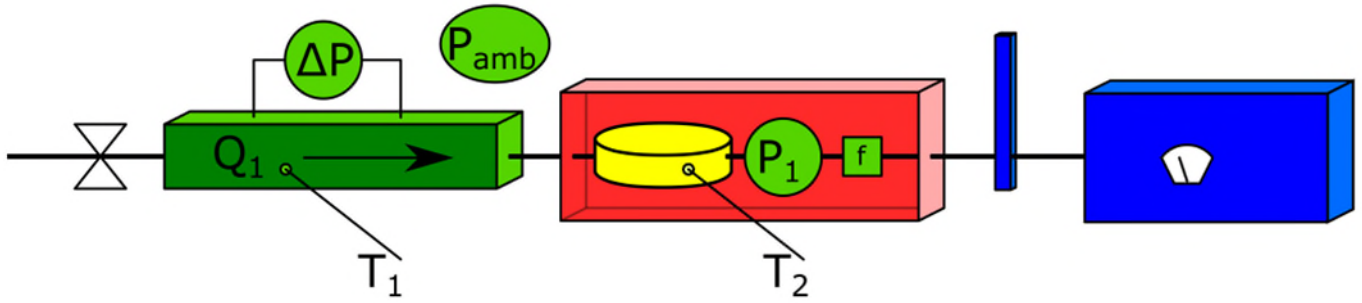
2. **Water ratio or fraction**
   - Traceable measurement of sample mass
   - Traceable measurement of sample volume or density

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

DTI Reference method for water content (EWV-DP)

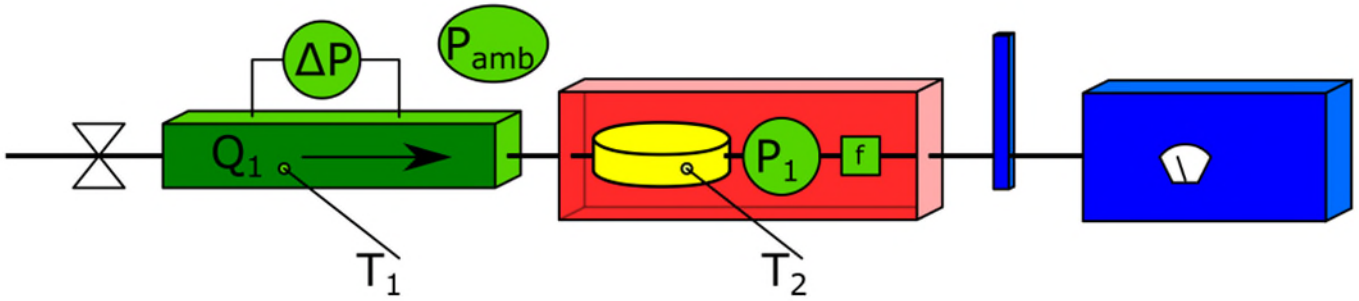
- Based on Evolved Water Vapour (EWV)
- Basic principle
  - Sample is dryed in flow of dry air
  - Measure
    - Humidity (= Evolved water vapour)
    - Air flow
  - Water content = Flow × Absolute humidity
  - Dry sample pragmatically defined as measured dewpoint < -15 °C
- Advantage: Specific water detection, Traceable
- Disadvantages: Single point (place, time), cumbersome, destructive

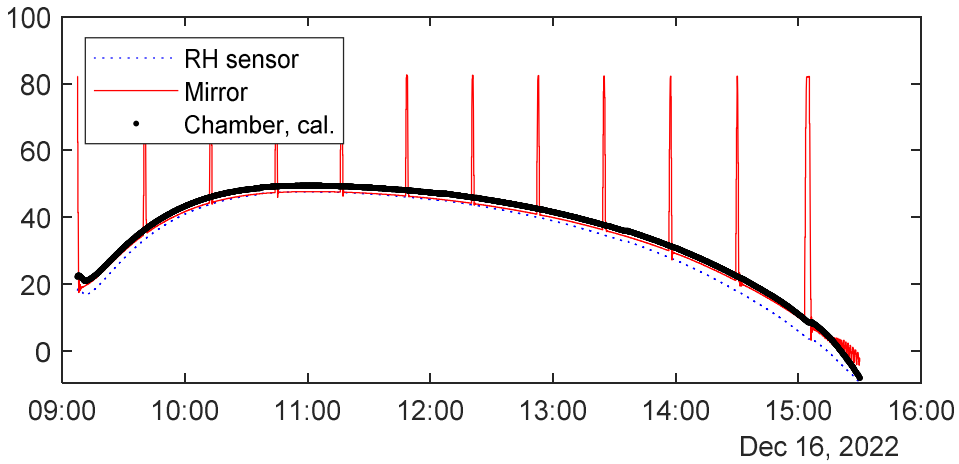


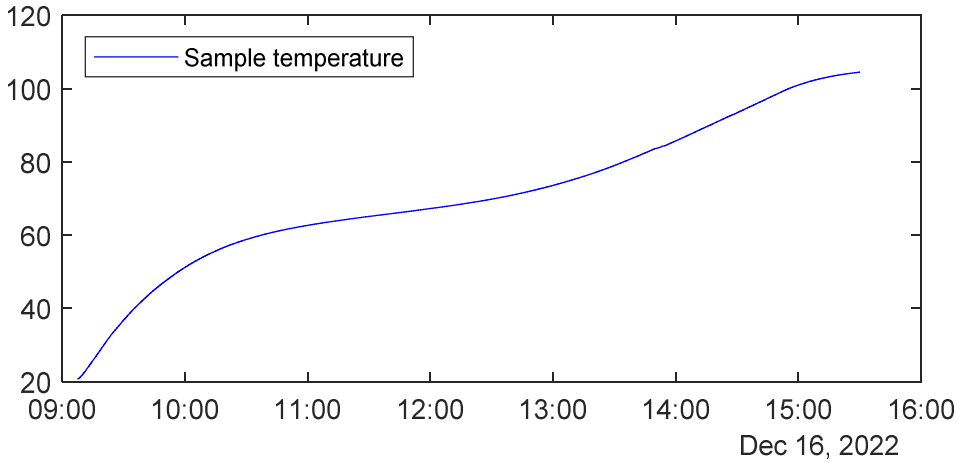
DANISH TECHNOLOGICAL INSTITUTE

SoMMet

DTI Reference method for water content (EWV-DP)







Status: Have been established and is being validated
With soil samples.

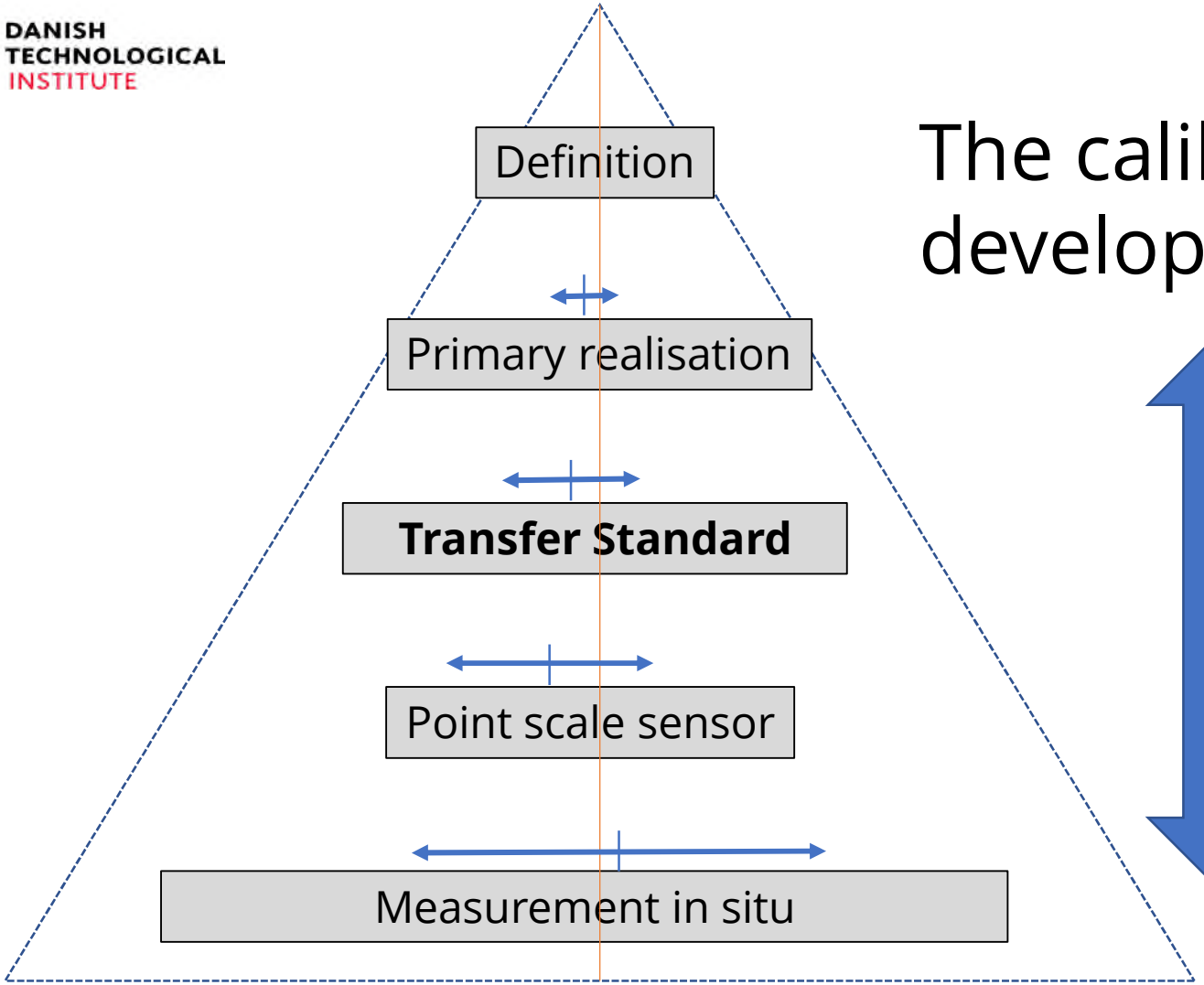
Expected uncertainty is < 1 – 1.5 % water mass fraction based on earlier experience using biomass samples (wood chips)[1]

1. EURAMET P1560 Interlaboratory comparison of moisture measurements with biomass samples

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

The calibration hierarchy developed in SoMMet



For every step in the chain of calibrations the uncertainty increases

Too low uncertainties ”breaks” the traceability chain

Objective: *To develop metrological framework, including primary and secondary transfer standards, to ensure SI-traceable point-scale soil moisture measurements with uncertainties of 5 % under laboratory conditions.*

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

A portable Loss on drying instrument for on-site calibration

Problem 1: Defining the dry state
- Water can be adsorbed or structural

Problem 2: Attaining a dry state
- Volatiles → mass loss
- Oxidation of matrix → mass increase
- Loss of organic matter → mass loss

Problem 3: What is "oven dry"?
- Drying time
- Lab relative humidity (→ water activity)
- Temperature distribution in oven

DANISH TECHNOLOGICAL INSTITUTE

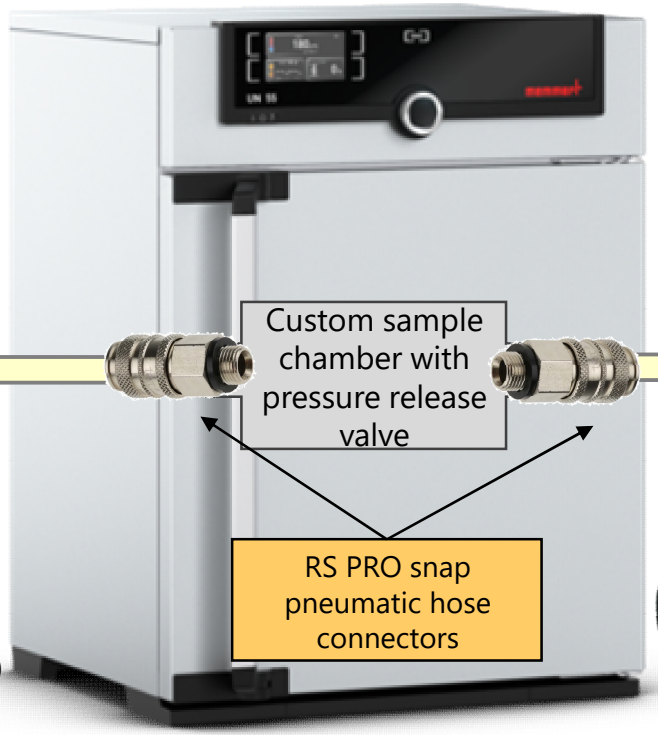
SoMMet

Development status

Oven: MEMMERT UN55 Plus

Compressor Jun-Air OF322-25B (air pumped to 12 bar pressure, dew point of expanded air at approx. -15 degrees celsius)

Swagelok flow limiter, needle valve (SS-SS4-VH)



Custom sample chamber with pressure release valve

RS PRO snap pneumatic hose connectors

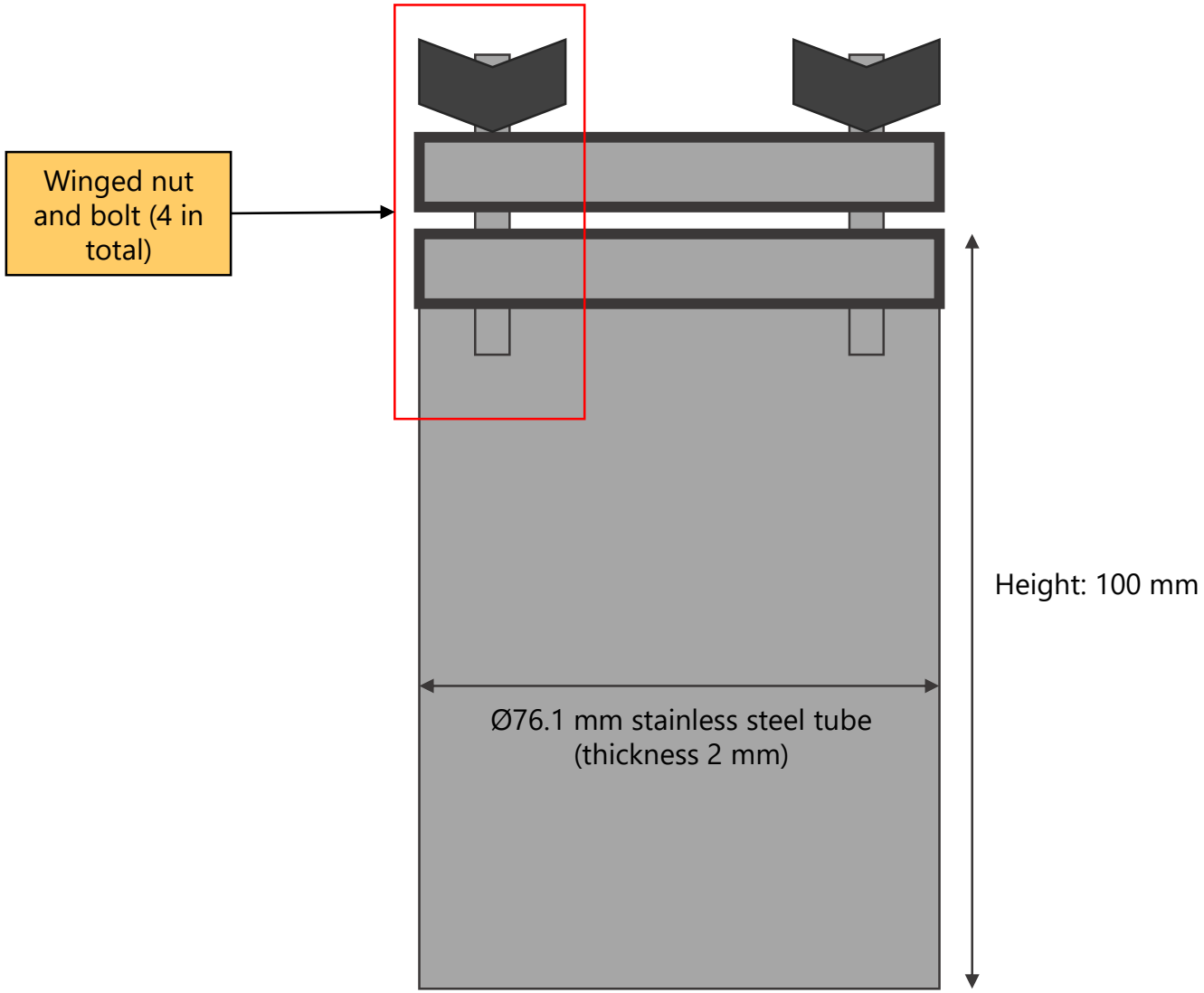
Vaisala HMP9 dew point sensor

Weight to calibrate mass scale (working range compatible with mass of sample chamber)

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

Sample chamber idea



DANISH TECHNOLOGICAL INSTITUTE

SoMMet

Conclusions and outlook

- A primary standard has been established and is being validated using soil-samples
- A secondary standard is being constructed and will be validated by comparison to the primary set-up using representative soil samples
- The volume of a soil-sampler will be calibrated
- The secondary standard can then be used on-site to ensure SI-traceability to the point-scale sensors used at the test sites.

DANISH TECHNOLOGICAL INSTITUTE

SoMMet

Co-funded by the European Union

European Partnership

METROLOGY PARTNERSHIP

EURAMET

The project 21GRD08 SoMMet has received funding from the European Partnership on Metrology, co-financed by the European Union's Horizon Europe Research and Innovation Programme and by the Participating States.