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Introduction to the BIOFMET project and the concept of metrological traceability

Workshop on metrology for biofuel industry
28-29 March 2023, PTB, Braunschweig, Germany

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Aim:

To optimize energy production based on solid and liquid biofuels through more accurate and faster determination of parameters impacting the calorific value (moisture, impurities, ash-content)

The project is interdisciplinary between thermal and chemical quantities

Objectives:

- To develop traceable online measurements for water content in solid biofuels,
- To develop improved methods for the sampling of biofuels
- To develop validated methods for the online measurement of ash content.
- To develop validated methods to determine the amount and nature of impurities in liquid biofuels
- To develop a traceable method for the online determination of the calorific value of liquid biofuels

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

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"

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- The Great Pyramid of Giza Built in the 26th century BC
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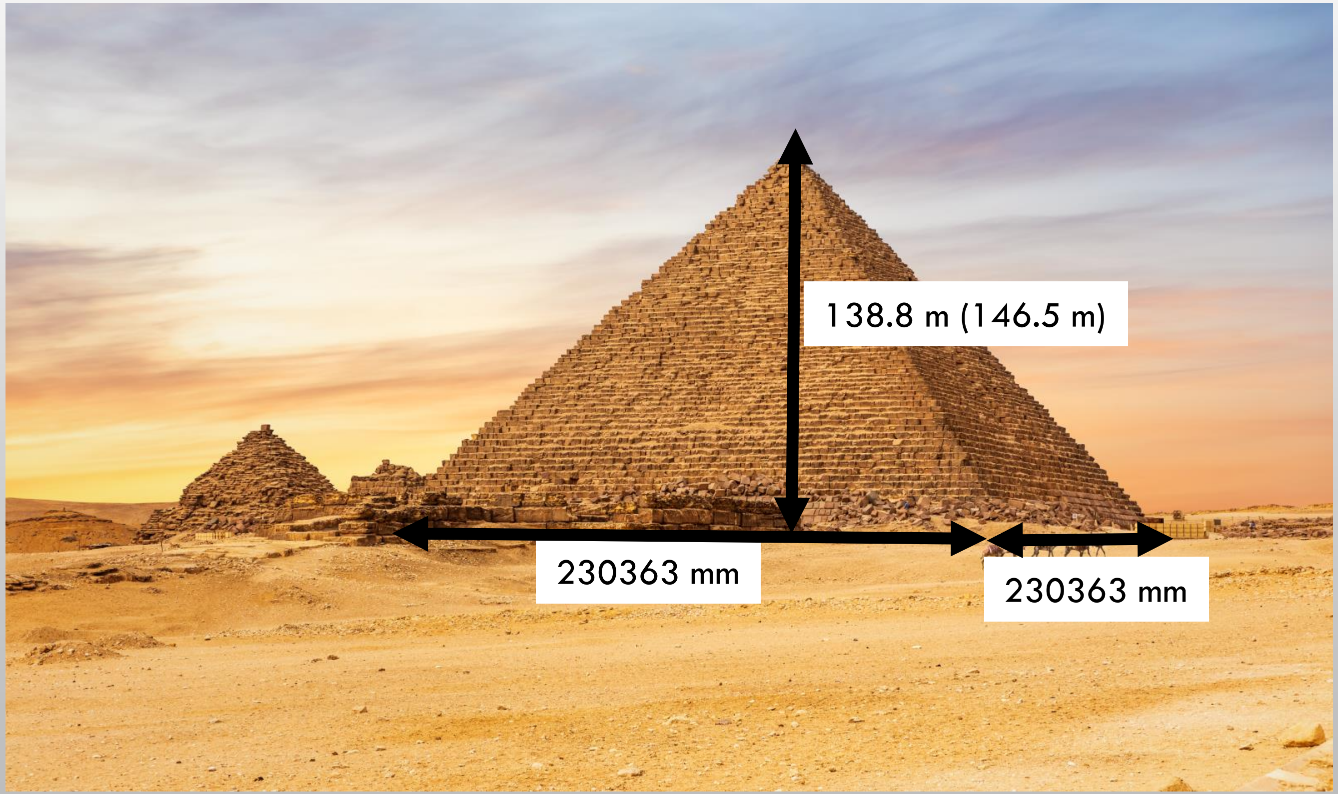
- Oldest and only existing of the "Seven Wonders of the Ancient World"

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



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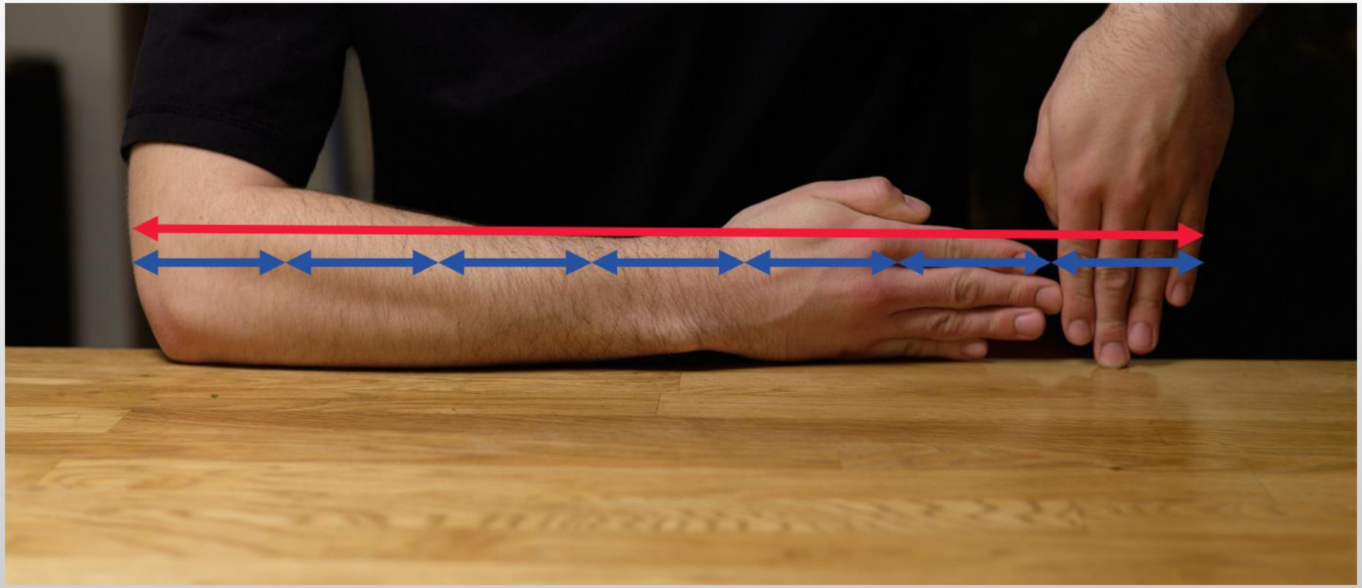
Step 1: Define a unit of length:



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

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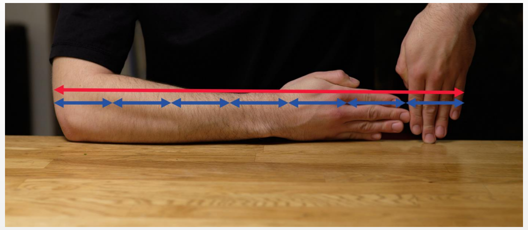

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Step 2: Realize the unit from its definition

https://collezioni.museoegizio.it/

Step 3: Make copies – and calibrate them by comparison

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

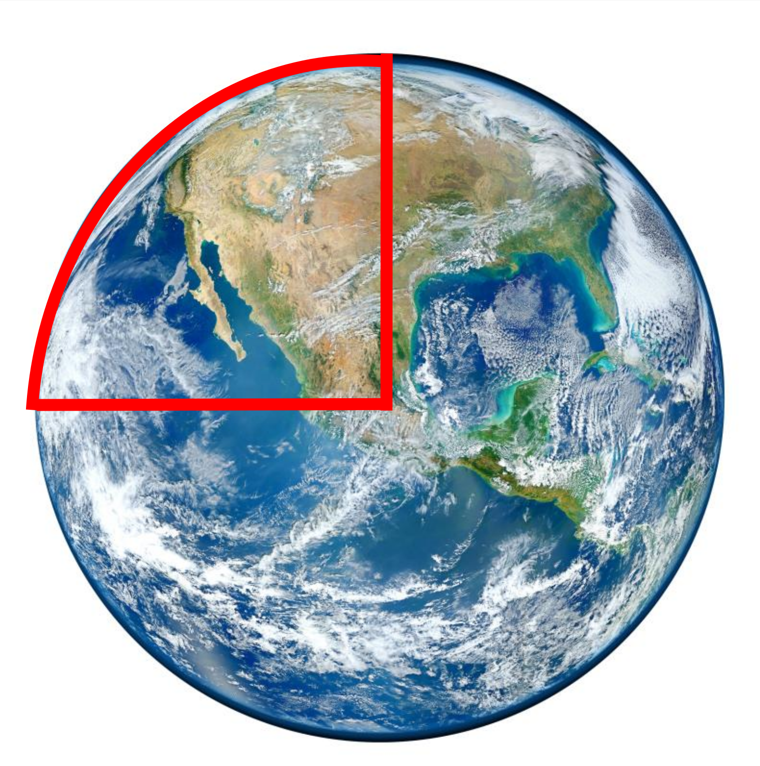
Traceability, calibration and quality control

The decree of the pharaoh is called the meter convention nowadays

In France in 1791 it was decided to define a new unit of length, the meter

1 meter was defined as 1/10,000,000 of the quarter meridian, the distance between the North Pole and the Equator along the meridian through Paris (a physical constant)

By astronomical measurements it was found that the distance from Dunkirk to Barcelona was about 1/10 of quarter meridian



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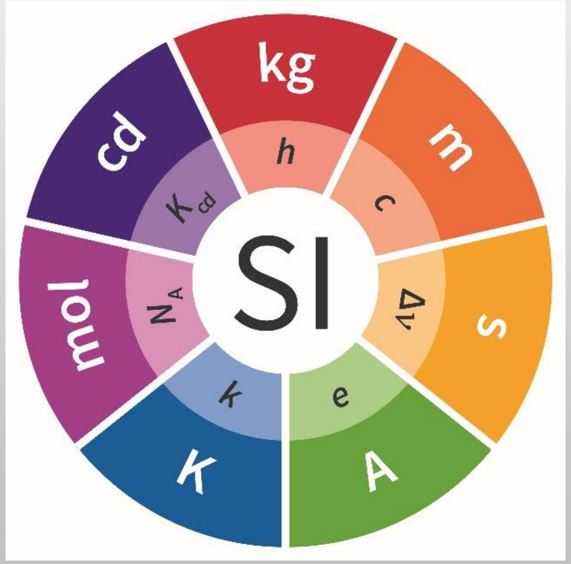
4 platinum rods (base measures) were made and the metrologists Jean Baptiste Joseph Delambre and Pierre Méchain, accurately measure the distance (lasting from 1792 to 1799)

A platinum rod was made that as accurately as possible was a 1/10,000,000 of the quarter meridian – a realisation of a meter was made.

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 x 10^{-34} J s
- the elementary charge e 1.602 176 634 x 10^{-19} C
- the Boltzmann constant k 1.380 649 x 10^{-23} J/K
- the Avogadro constant N_A 6.022 140 76 x 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.

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Metrological Traceability

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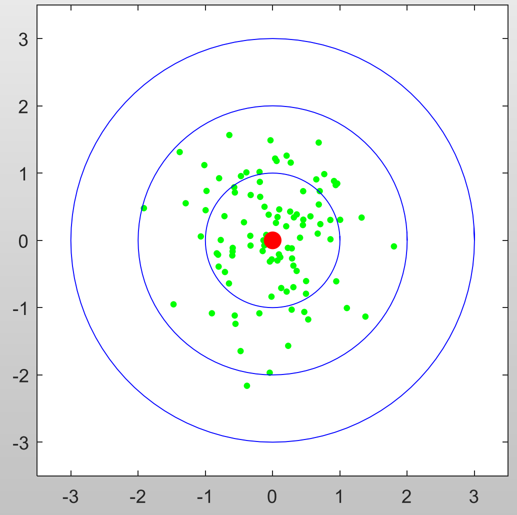
What is measurement uncertainty?

Precision or accuracy



Good accuracy
Poor precision

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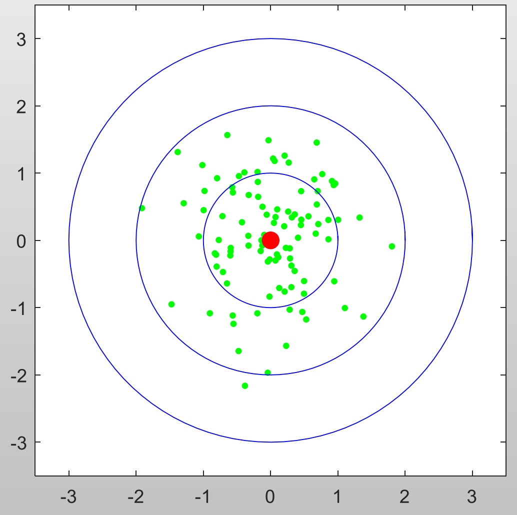
Precision or accuracy



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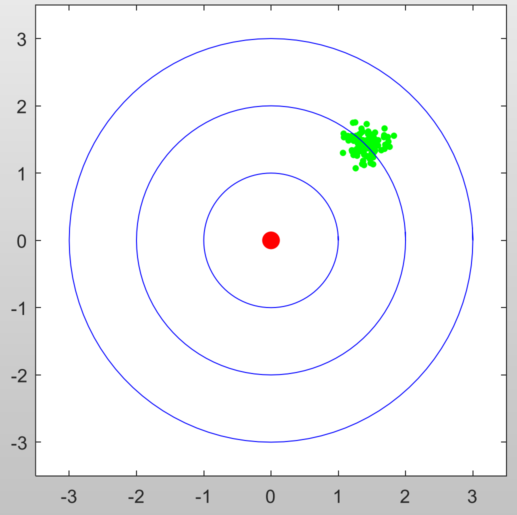

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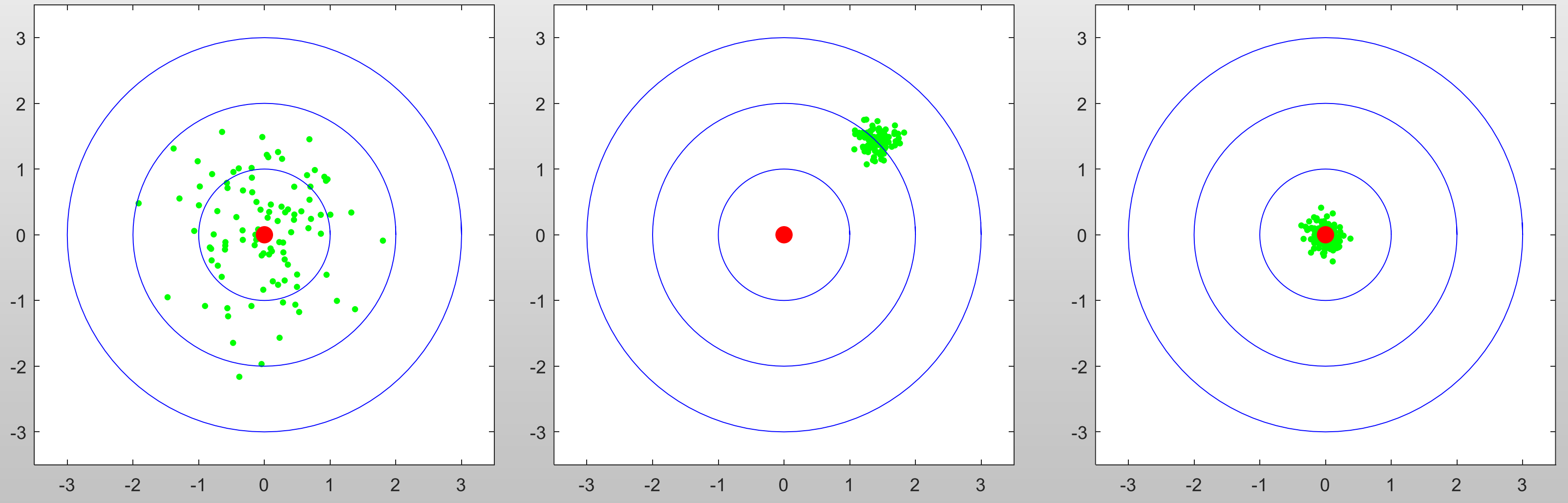

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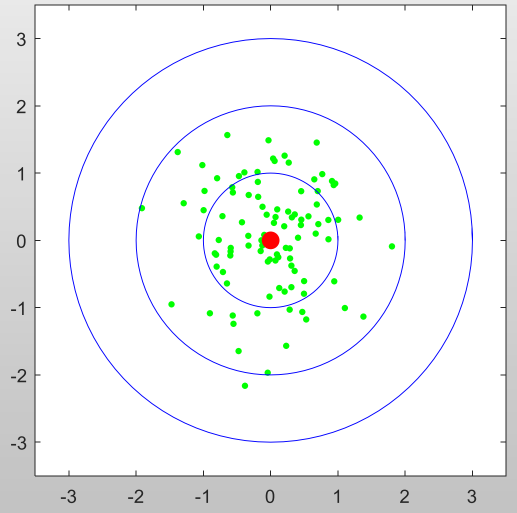

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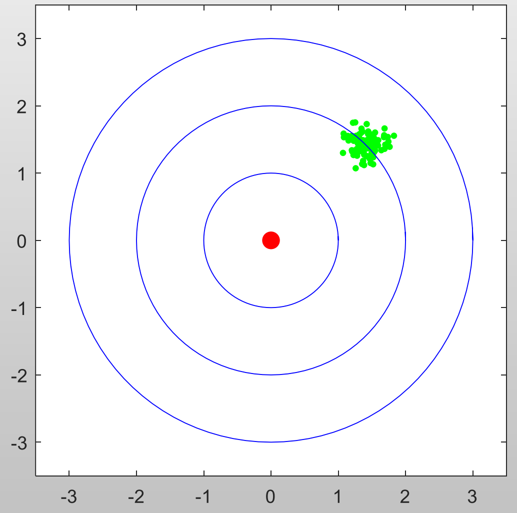

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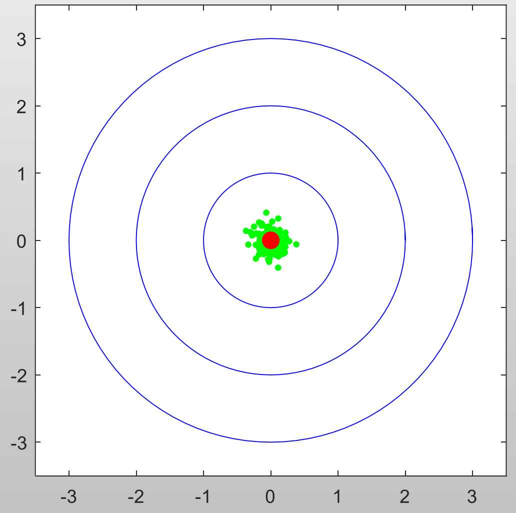

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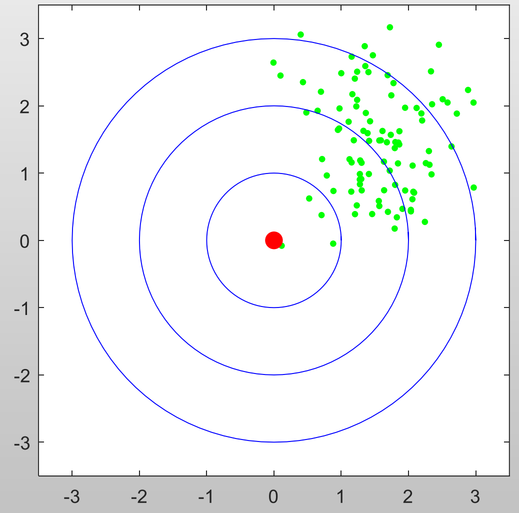

Poor accuracy
Poor precision

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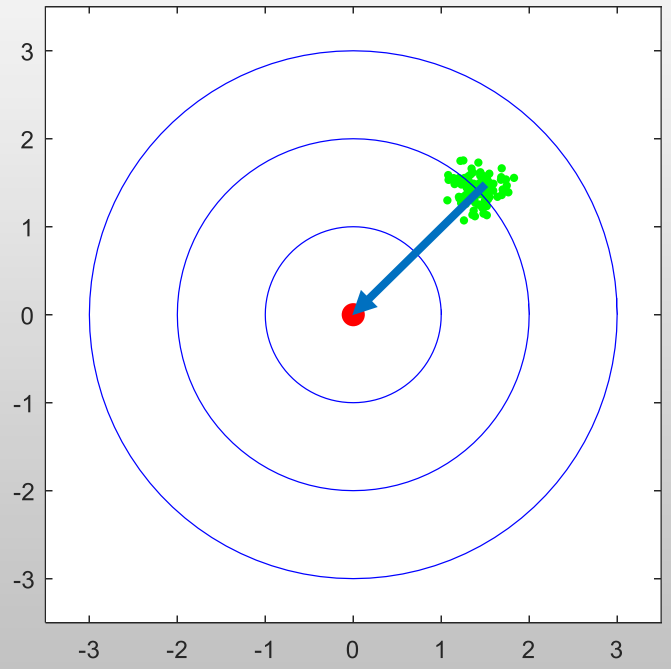
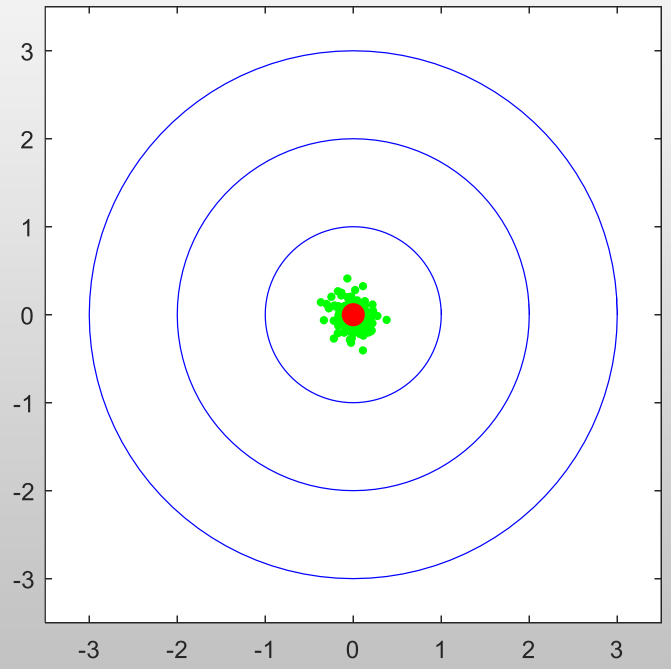
Solution: calibration

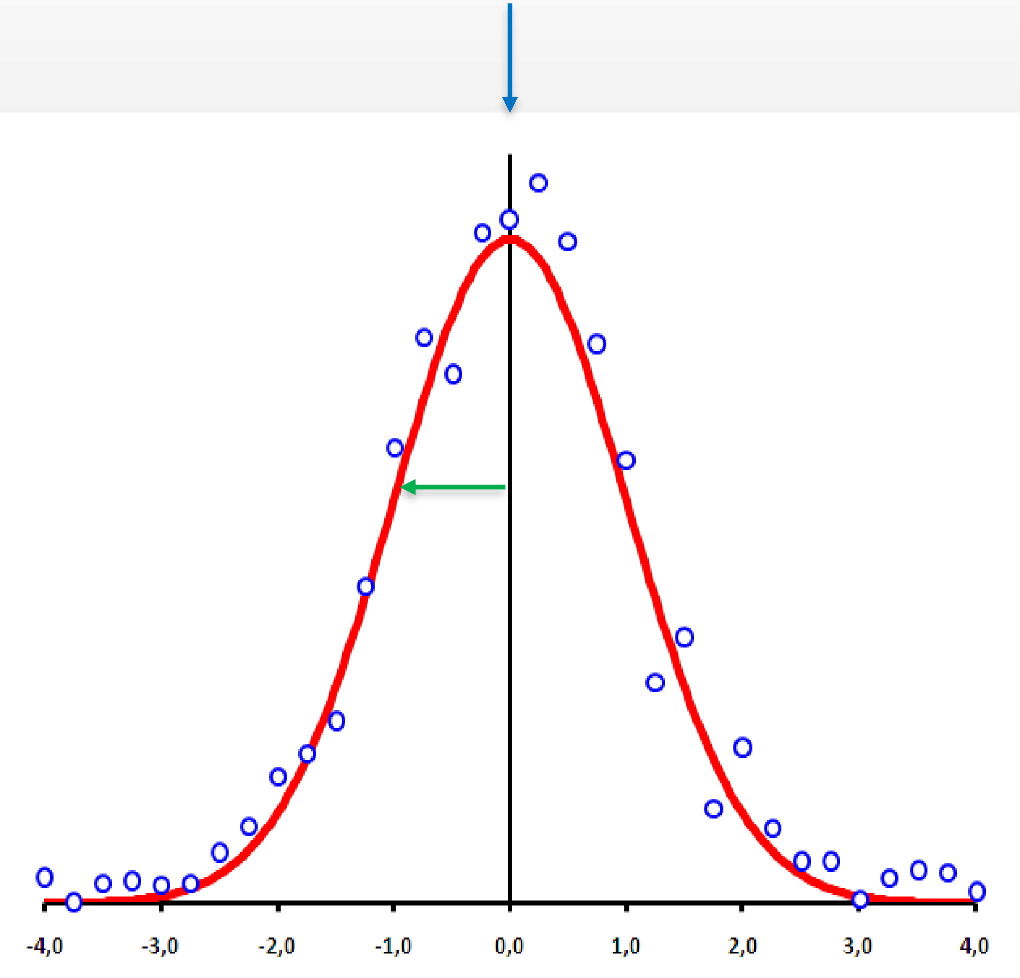




Determine systematic errors by calibration and correct the result

What is measurement uncertainty?

- parameter characterizing the dispersion of the quantity values being attributed to a measurand (the mean value)



Uncertainty of calibration

- By calibrating a measurement instrument the error with respect to a reference is found
- This measurement has an uncertainty

Reference value °C	Reference value %rh	Indication %rh	Error %rh	Uncertainty %rh
0.08	61.62	56.50	-5.12	0.73
24.97	24.70	23.70	-1.00	0.21
25.01	60.79	55.70	-5.09	0.40
25.02	90.85	83.40	-7.45	0.57
50.17	60.70	56.65	-4.05	0.74

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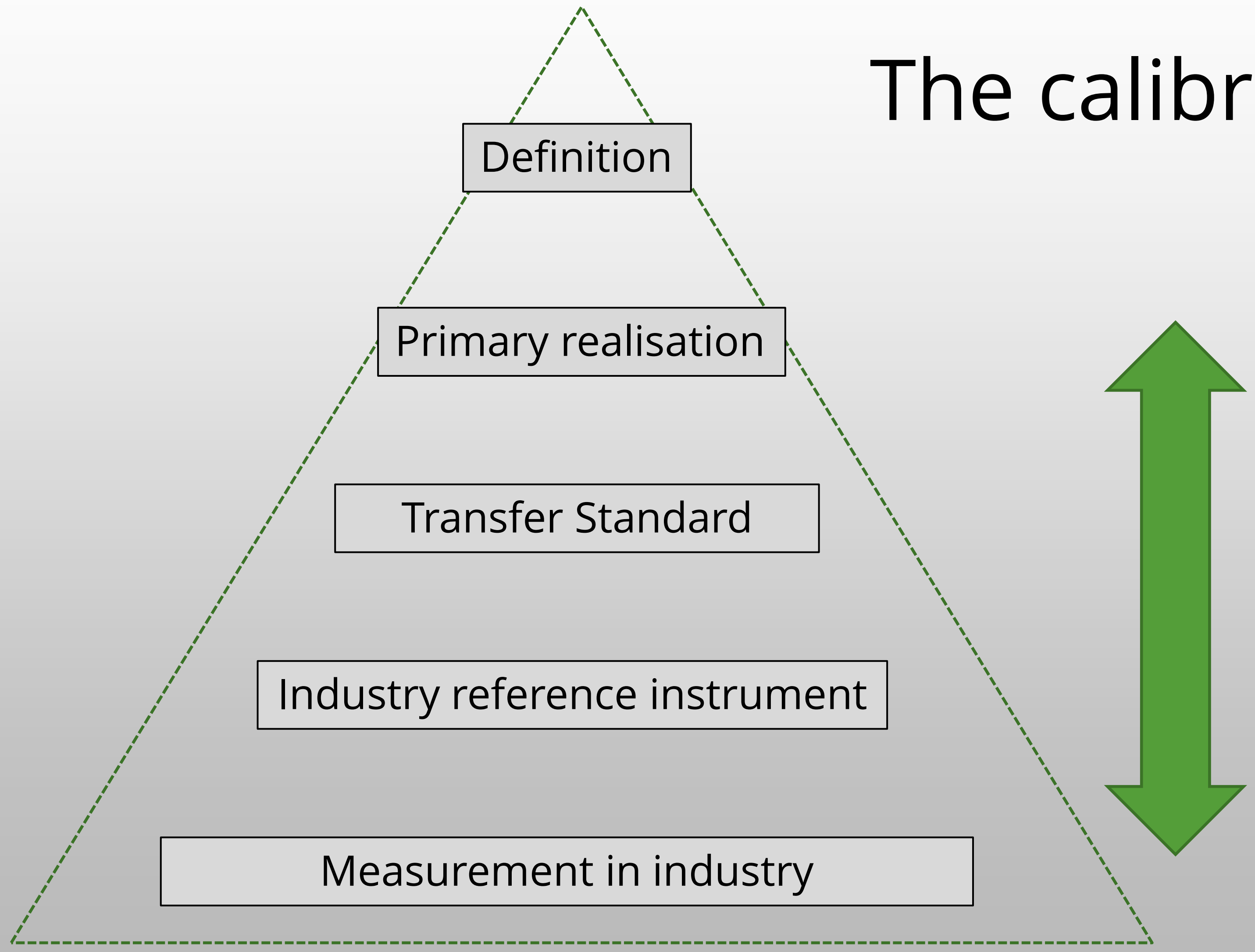
The calibration hierarchy



For every step in the chain of calibrations the uncertainty increases

Too low uncertainties ”breaks” the traceability chain

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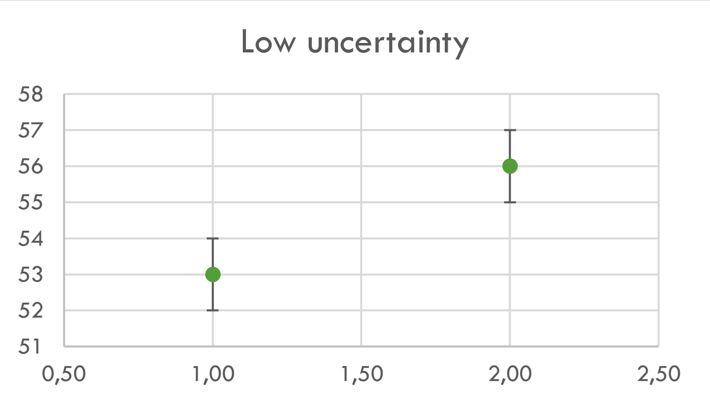
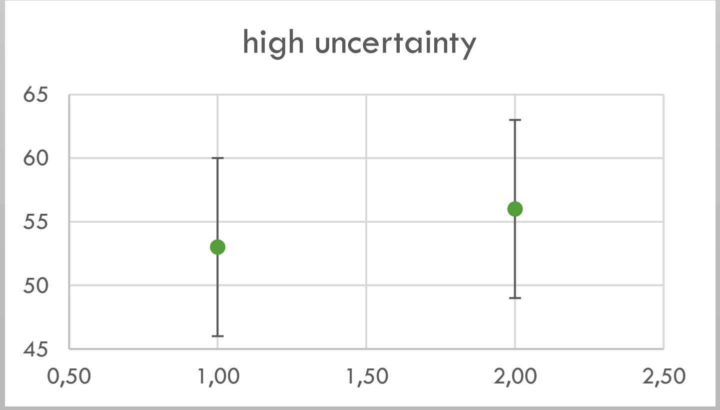
Measurement uncertainty – Why?

- Why don't we drop the measurement uncertainty?
- Alice drives 53 km/h
- Bob drives 56 km/h
- Who runs the fastest?
  - If Alice drives 53 ± 1 km/h and Bob drives 56 ± 1 km/h
  - If Alice drives 53 ± 7 km/h and Bob drives 56 ± 7 km/h
- If the uncertainty is high it is difficult to conclude differences between measurements.





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ISO 17025:2017 requirements

6.4.5: The equipment used for measurement shall be capable of achieving the mesurement accuracy and/or measurement uncertainty required to provide a valid result

How do we verify this?

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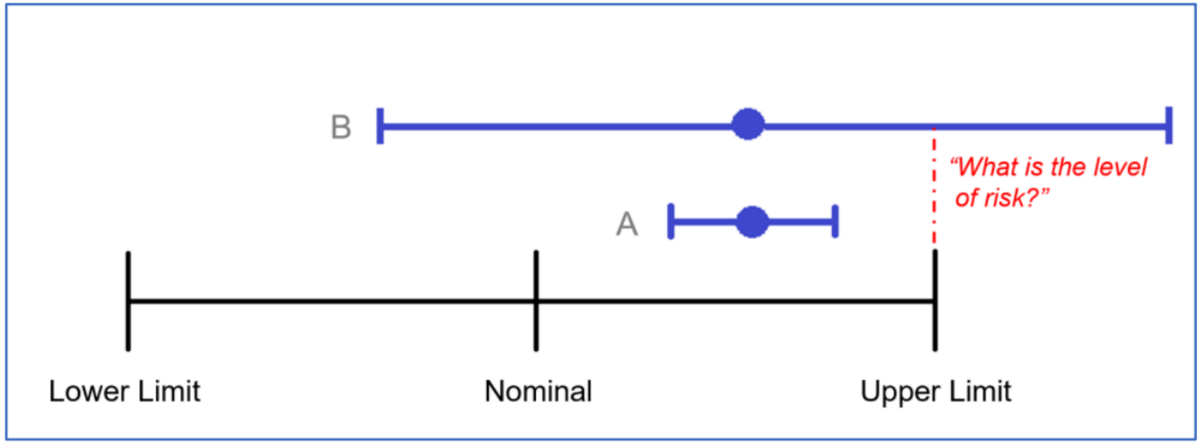
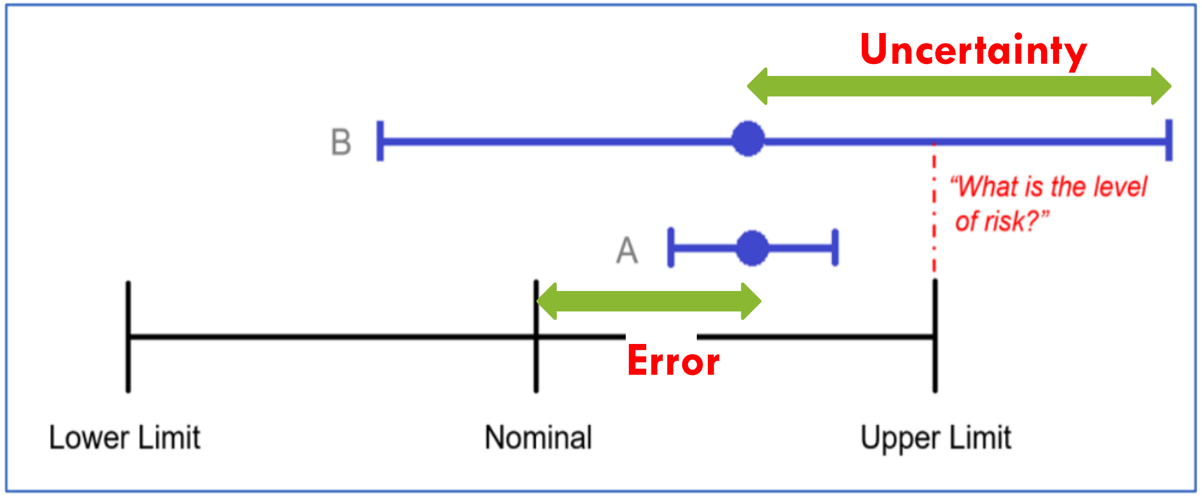


Figure 1. Illustration of Measurement Decision Risk



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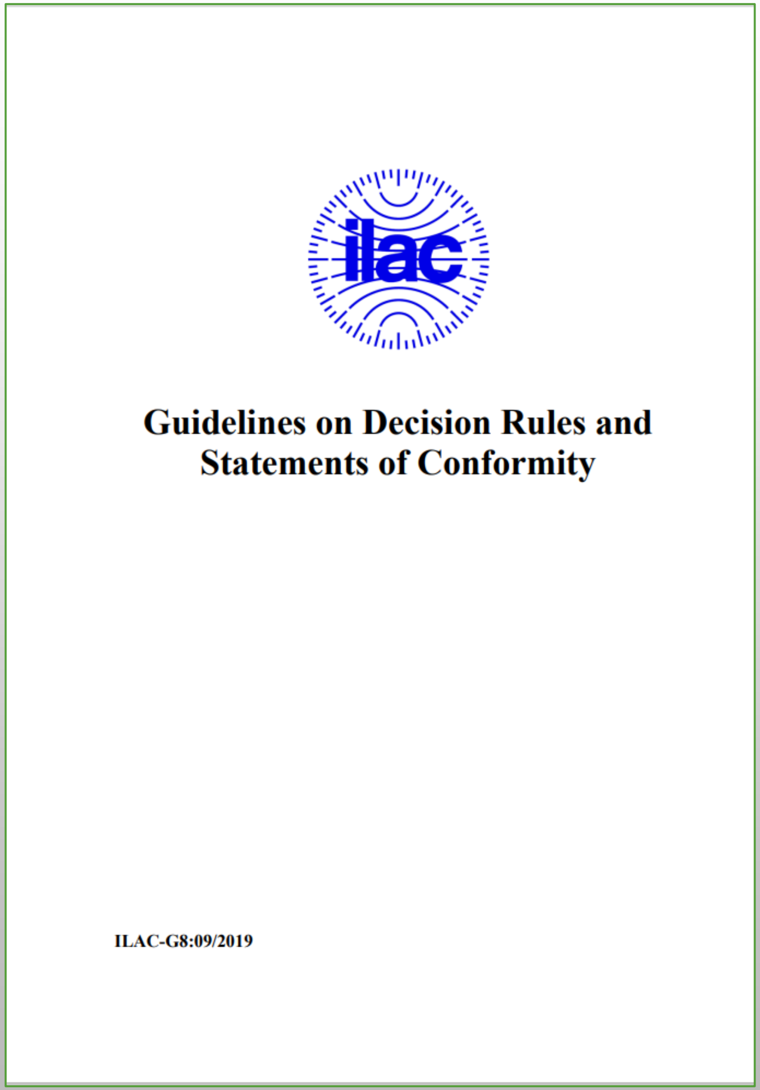
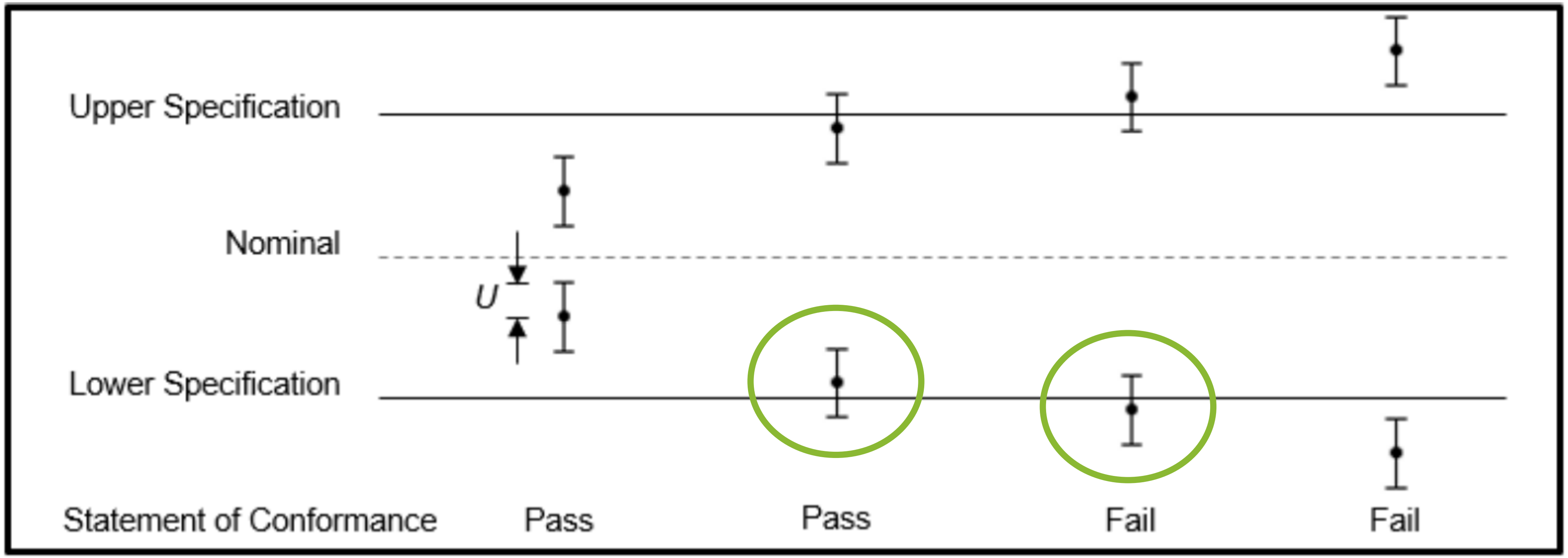

ilac

Guidelines on Decision Rules and Statements of Conformity

ILAC-G8:09/2019

One method (out of 4) from ILAC G9, where there is a risk of false acceptance

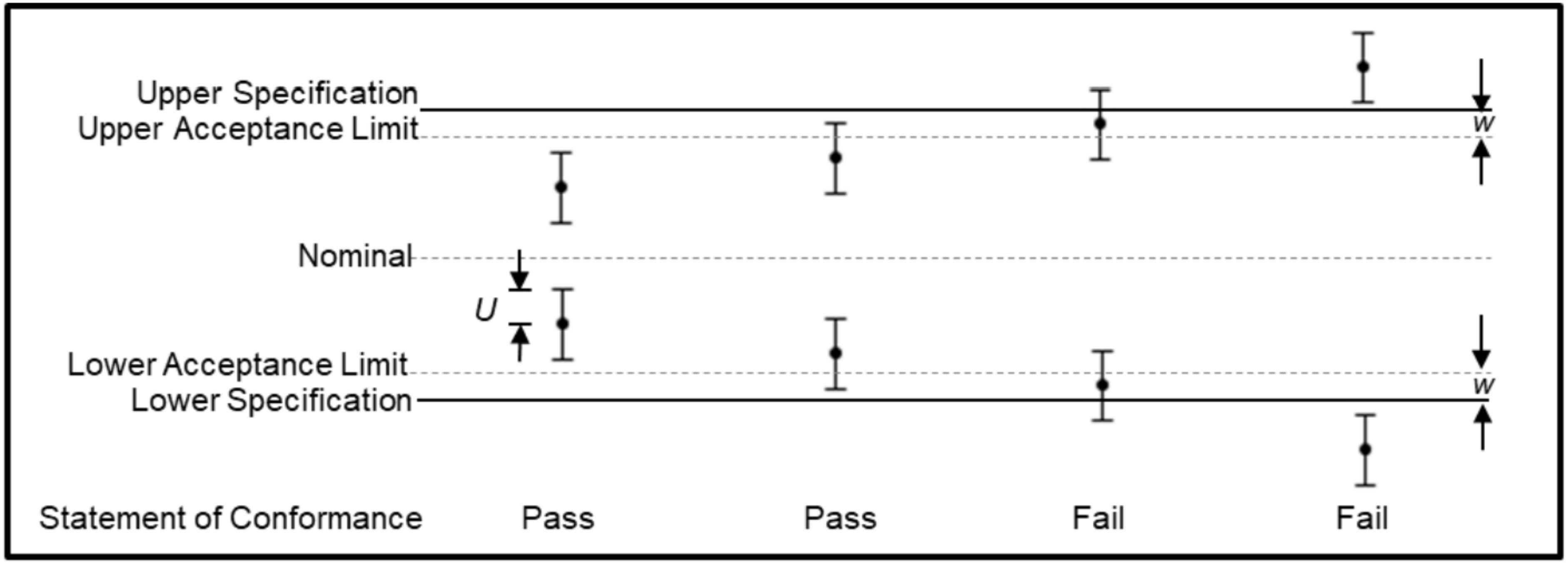


U = 95% expanded measurement uncertainty

Figure 3 Graphical representation of a Binary statement - Simple Acceptance

How do we control the risk?

One method (out of 4) from ILAC G9, where measurement uncertainty is taken into account



U = 95% expanded measurement uncertainty

Figure 4 Graphical representation of a Binary statement with a guard band

Which Guard-band (w) should one choose?

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Decision rule	**Guard band w**	**Specific Risk**
6 sigma	3 *U*	< 1 ppm PFA
3 sigma	1,5 *U*	< 0.16% PFA
ILAC G8:2009 rule	1 *U*	< 2.5% PFA
ISO 14253-1:2017 [5]	0,83 *U*	< 5% PFA
Simple acceptance	0	< 50% PFA
Uncritical	-*U*	Item rejected for measured value greater than $AL = TL + U$ < 2.5% PFR
Customer defined	*r U*	Customers may define arbitrary multiple of *r* to have applied as guard band.

Table 1. PFA – Probability of False Accept and PFR – Probability of False Reject
(Assumes a single sided specification and normal distribution of measurement results)

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

- Measurement uncertainty ensures that a measurement result is related to a reference on a “higher level” that in the end is compared with a primary realization of the unit – measurement uncertainty is a measure of the quality of a measurement.

- Thus, traceability is needed in order to make trustworthy measurements on all levels independent of method or instrument type.

This is what the BIOFMET project is trying to solve for biofuels measurements…

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Achievements

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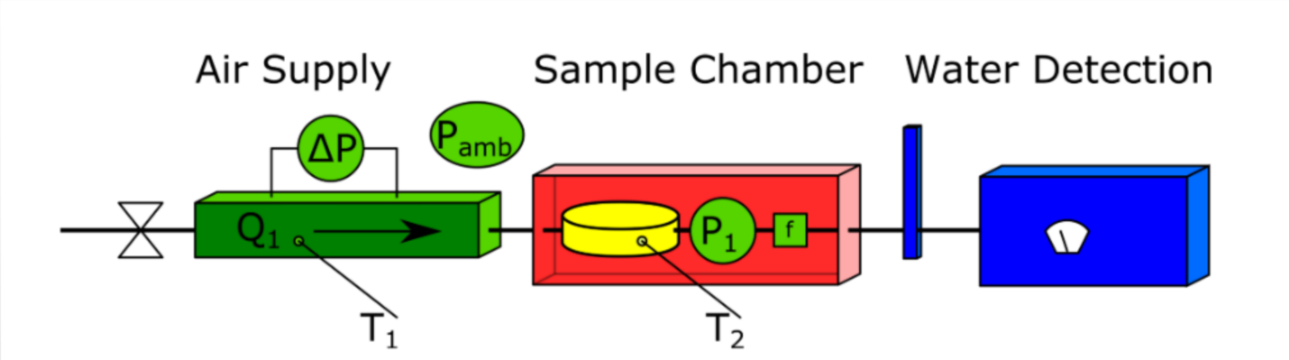
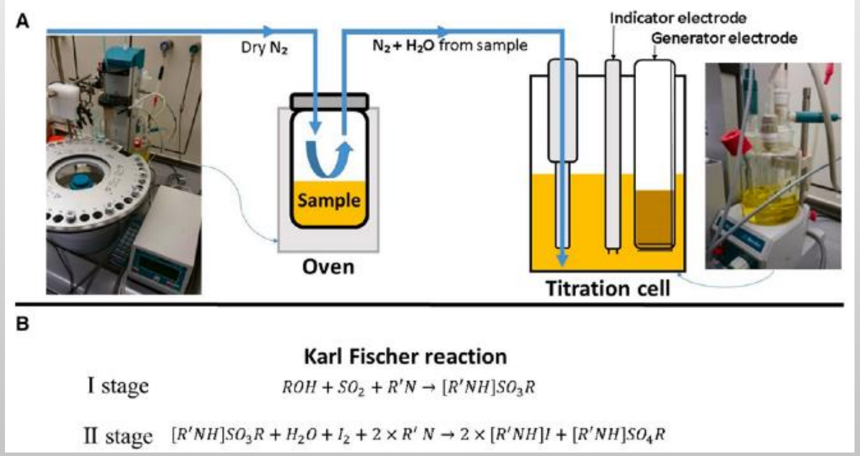
Underpinning metrology





- Traceable energy content measurements (calorific value)
- Traceable methods for the determination of impurities and residuals
- Calibration facility for moisture transfer standards

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The calibration hierarchy

- Definition
- Primary realisation
- Transfer Standard
- Industry reference instrument
- Measurement in industry

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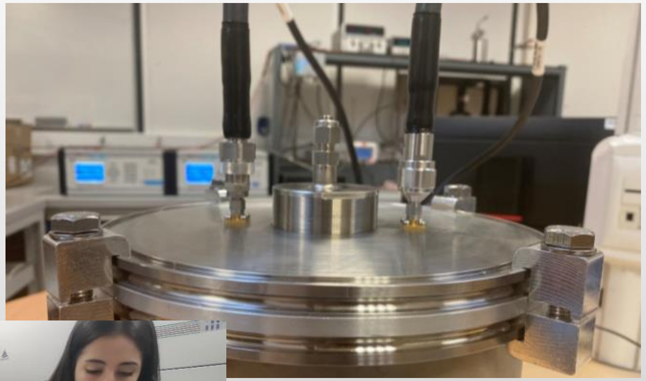
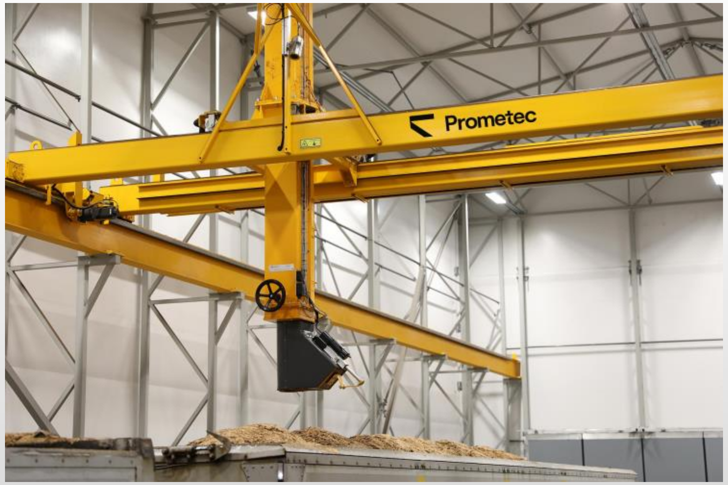
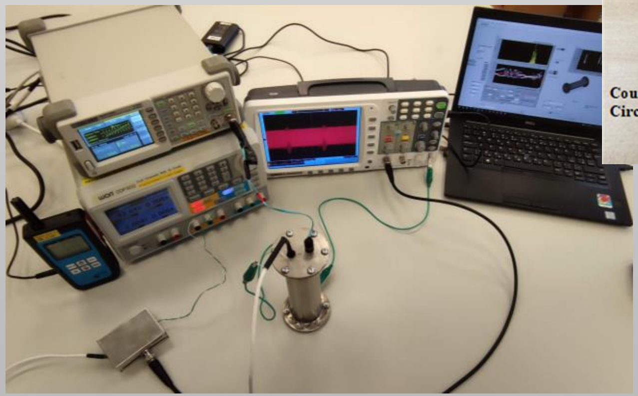
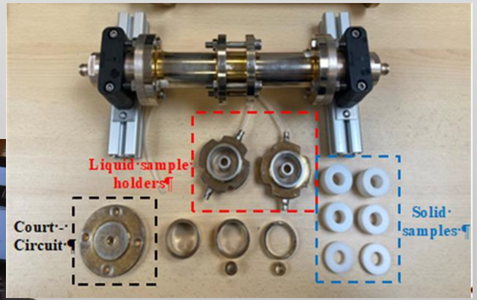
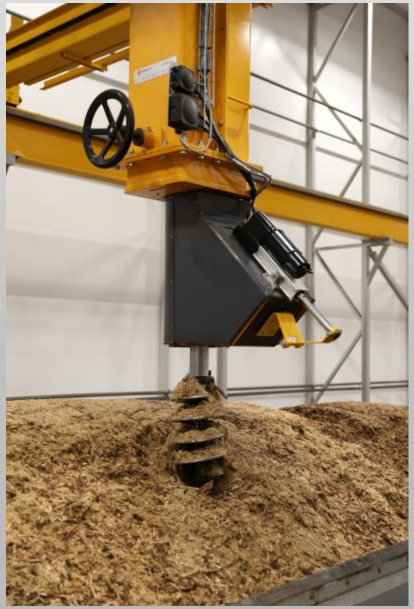
Development of online traceability







- Reference materials
- New transfer standard
- Sampling strategies

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The calibration hierarchy

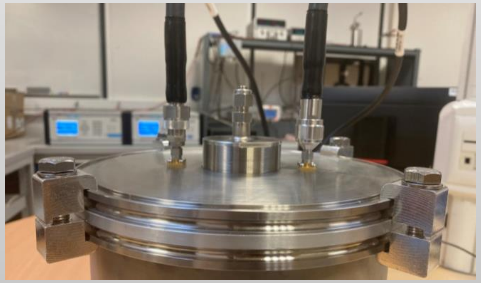
Definition

Primary realisation

Transfer Standard

Industry reference instrument

Measurement in industry



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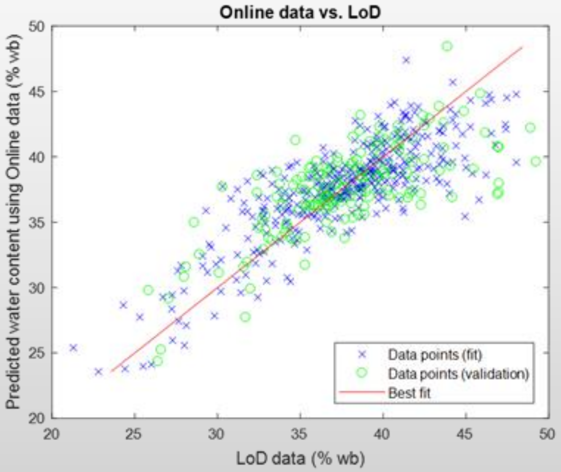
Industrial validation



The calibration hierarchy

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Primary realisation

Transfer Standard

Industry reference instrument

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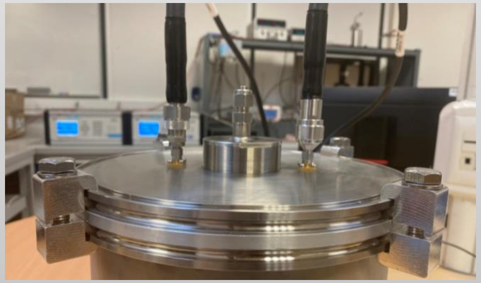

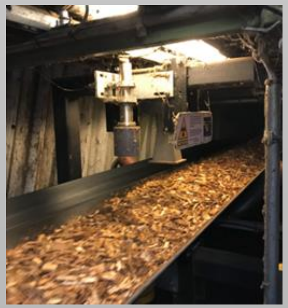

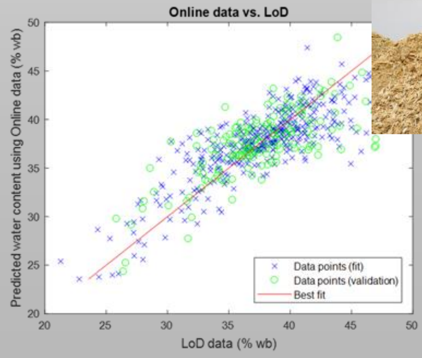

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Summary of the project

The key targets to be reached by the end of this project (and to be exploited in the 5 years that follow the end of the project) are as follows:

- Calibration methods and services are available for industry that ensures traceable on-line measurements for water and ash content in biofuels
- New methods, reference materials and services are available for determining the amount and level of impurities in liquid biofuels
- New methods for sampling of biofuels have been researched, validated and demonstrated and new automatic sampling devices for “representative sampling” is available on the marked.

This project will be considered a success if these targets are met and take up of the results has been demonstrated by standards developing organisations and end users.

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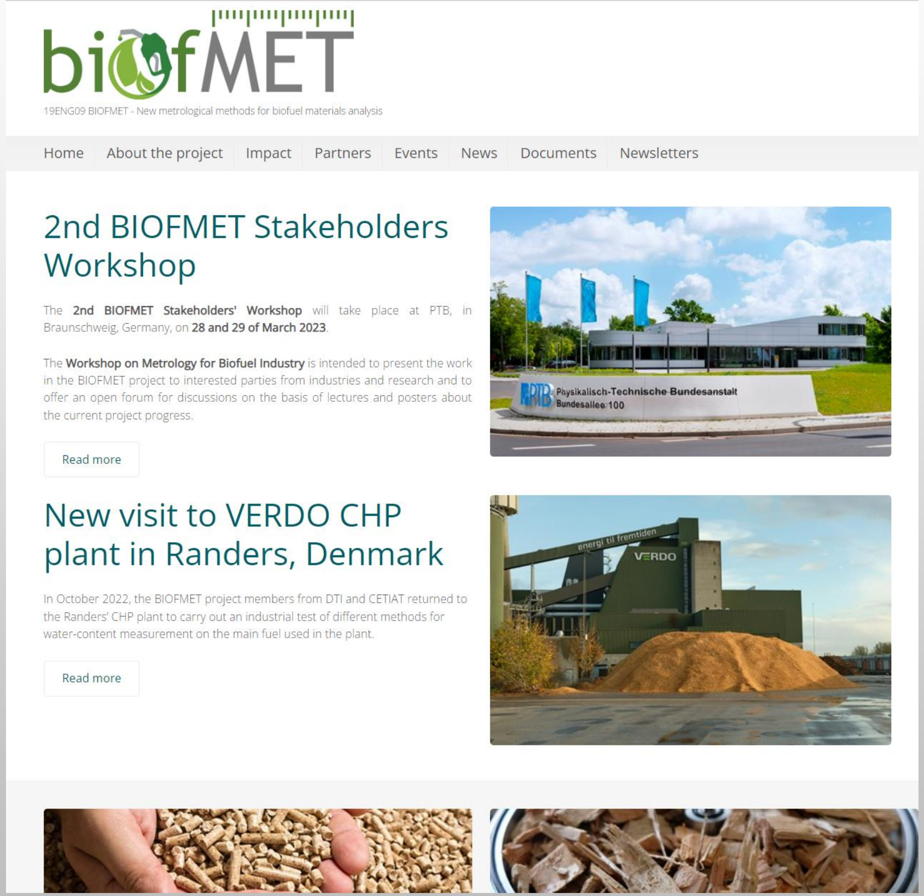

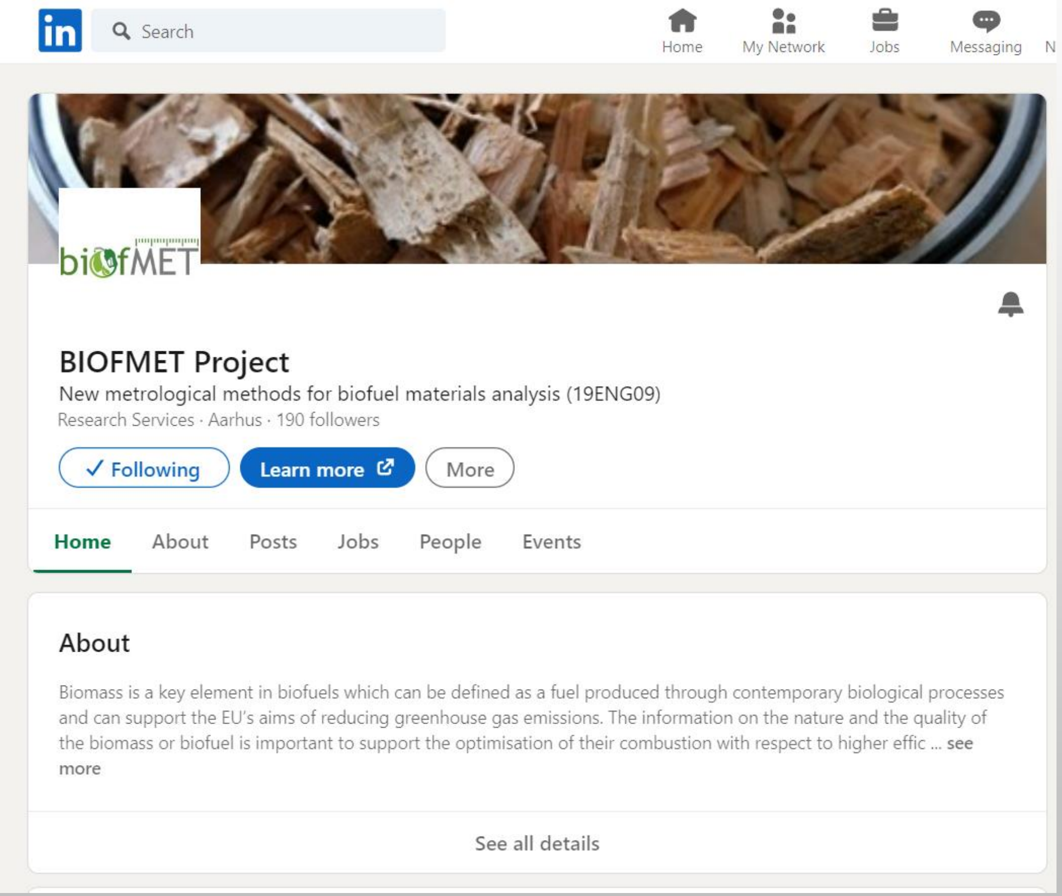

http://www.biofmet.eu/

https://www.linkedin.com/company/biofmet/

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Thank you for your attention!

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