SOLAR HEATING & COOLING PROGRAMME
INTERNATIONAL ENERGY AGENCY

# Round robin test on thermal conductivity/thermal diffusivity

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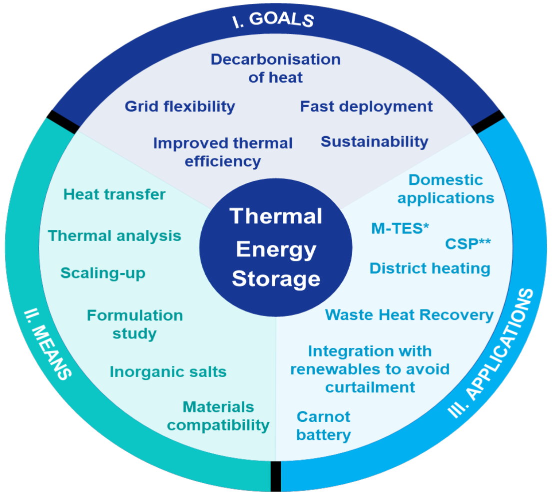
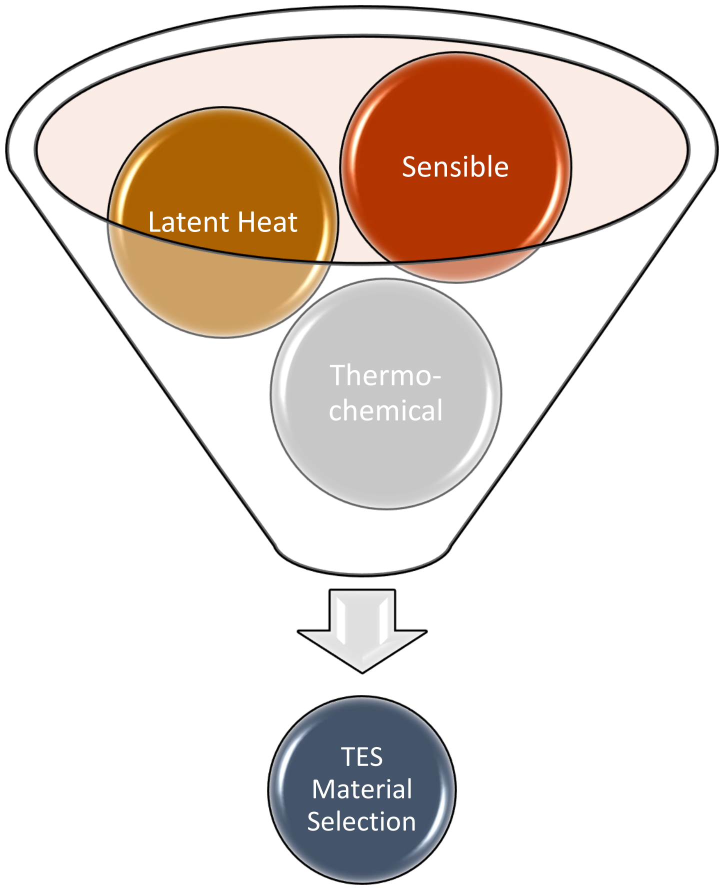
# Outline

- Motivation
- Thermal Conductivity/Thermal Diffusivity
- Round Robin test
    - RRT-Task A1.1
    - RRT-Data Collection
    - RRT-Procedures
    - RRT-Data Analysis
- Challenges




# Motivation

## TES system design




- ✓ Physical and technical requirements:
  - ✓ Small volume change
  - ✓ High energy density
  - ✓ Small or no supercooling
  - ✓ No phase segregation
  - ✓ Low vapour temperature
  - ✓ Chemical and physical stability
  - ✓ Compatible with other materials
  - ✓ **Good thermal conductivity**
  - ✓ Cost

# Thermal Conductivity/Thermal Diffusivity

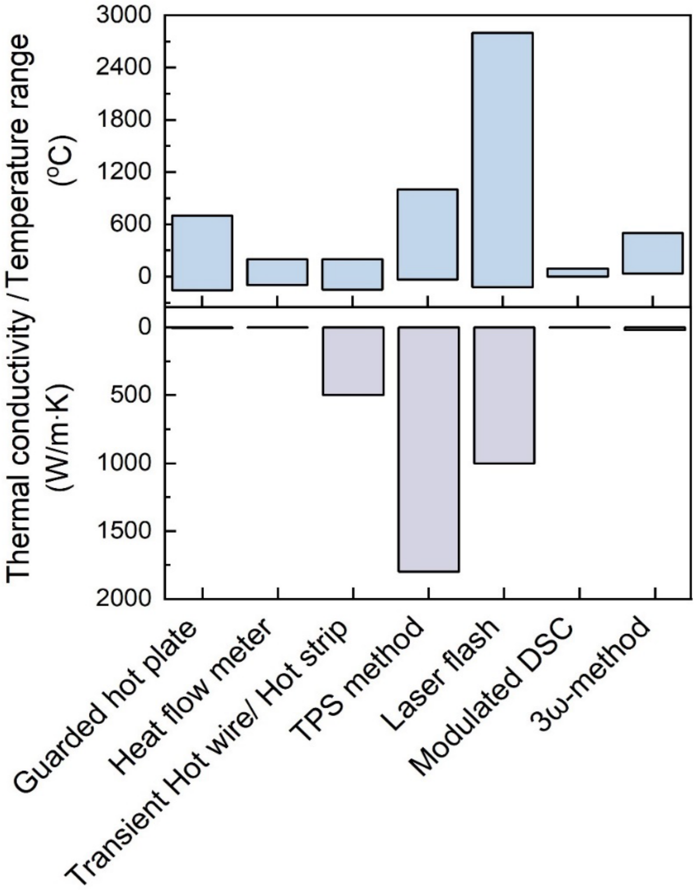



Fig 1. Thermal conductivity and temperature measurement range for steady-state methods and transient methods.

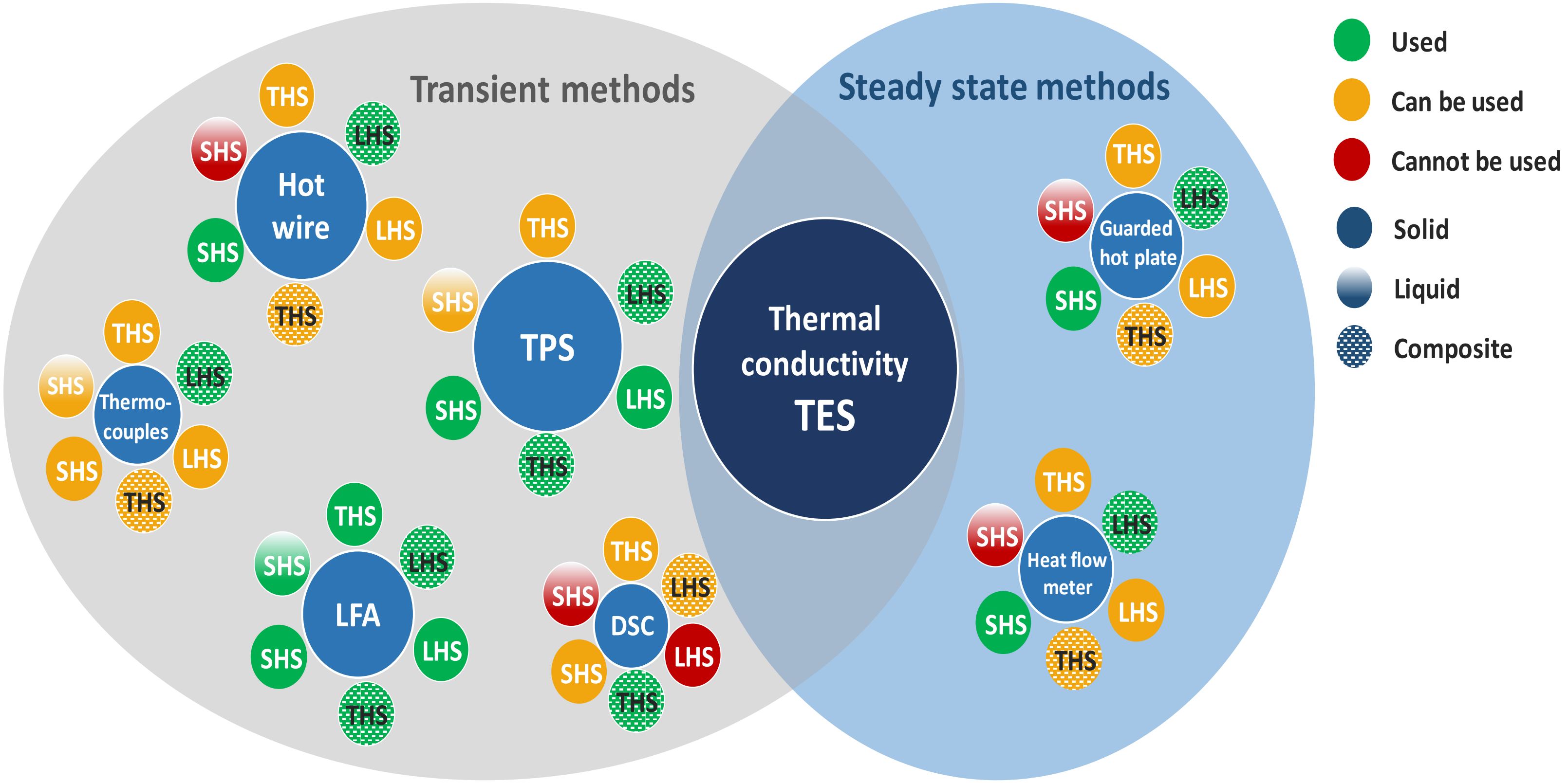


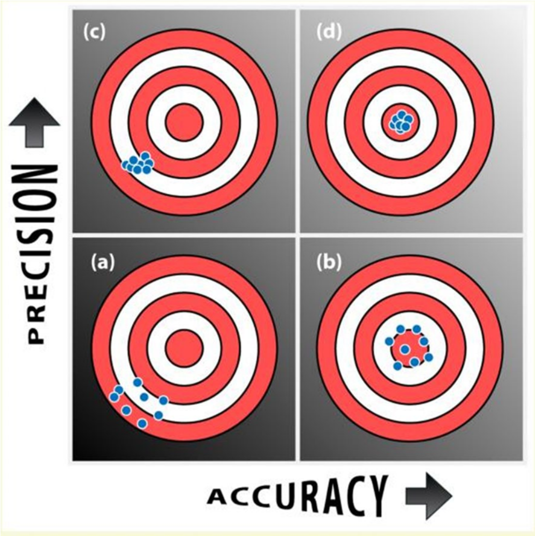

*Fig 2. Thermal conductivity/diffusivity methods used for different TES materials. SHS ( Sensible heat storage), LHS (Latent heat storage) and THS (Thermochemical storage).*



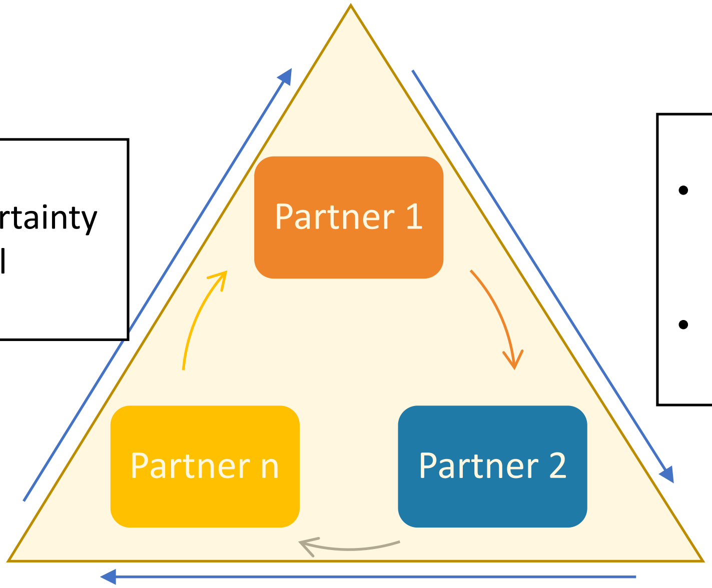
*Anabel Palacios, Lin Cong, M.E. Navarro, Yulong Ding, Camila Barreneche, Thermal conductivity measurement techniques for characterizing thermal energy storage materials – A review, Renewable and Sustainable Energy Reviews, Volume 108, 2019, Pages 32-52, https://doi.org/10.1016/j.rser.2019.03.020.*

# Round Robin Test

- RRT-interlaboratory test (measurement, analysis, or experiment) performed independently several times
  - ➢ Develop standardized measurement procedures for thermal energy storage materials based on round robin tests for several characteristics, thermal conductivity/thermal diffusivity




**Analysis**
- Identification of uncertainty supported by minimal information table




**Process**
- Collection of descriptive information and quality criteria into minimal information tables
- Development of SOP

**Measurement**
- Thermal conductivity
- Thermal diffusivity



*Elberskirch, L.; Sofranko, A.; Liebing, J.; Riefler, N.; Binder, K.; ... ins, R.P.F.; et al. How Structured Metadata Acquisition Contributes to the Reproducibility of Nanosafety Studies: Evaluation by a Round-Robin Test. Nanomaterials **2022**, 12, 1053. https://doi.org/10.3390/nano12071053*

# Round Robin Test

Organization → Transfer → Measurement → Transfer → Analysis

**Process**

**RRT design**
- Description
- Equipment
- Material
- SOP development
  - -Methods
  - -Procedures
- Participants

Collaboration

**Distribution**
- Materials

**Curation**
- Requirements
  - Descriptive information
  - Quality criteria

Partner 1

Partner 2

Partner n

Collaboration

**Sharing**
- Raw data
- Filled MIT

**Data management**
- Review of request
- Corrective actions

**Analysis**
- Raw data

**Statistics**
- Evaluation of raw data
- Uncertainty analysis
- Identification of deviations by MIT

**MIT**

**Tables**
- Participants
- Equipment
- Materials (sample preparation)
- SOPs
- Results
- Analysis and statistics

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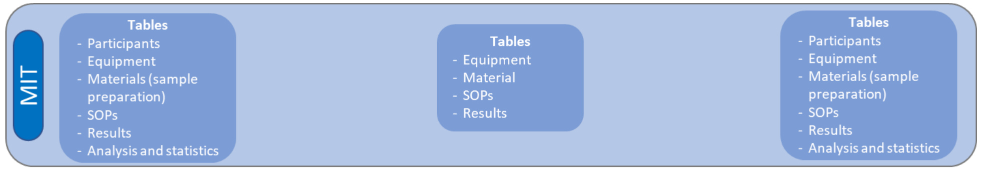
AIT Austrian Institute of Technology; CAE zero carbon; University of Birmingham; HSLU Lucerne University of Applied Sciences and Arts; Fraunhofer; Northumbria University Newcastle; CIC energiGUNE; Universidad Zaragoza; Université d'Artois; DLR; CanmetÉNERGIE Varennes CanmetENERGY; Universitat de Barcelona; Çukurova Üniversitesi; SINTEF; Consiglio Nazionale delle Ricerche; Universidad del País Vasco Euskal Herriko Unibertsitatea

MIT: minimum information table that specifies necessary minimum information to be provided along with experimental in a flexible and modular manner.



*Elberskirch, L.; Sofranko, A.; Liebing, J.; Riefler, N.; Binder, K.; Bonatto Minella, C.; Razum, M.; Mädler, L.; Unfried, K.; Schins, R.P.F.; et al. How Structured Metadata Acquisition Contributes to the Reproducibility of Nanosafety Studies: Evaluation by a Round-Robin Test. Nanomaterials* ***2022****, 12, 1053. https://doi.org/10.3390/nano12071053*




# RRT-Task A1.1

## Participants
- Research center
- Contact person

## Equipment
- Instrument information
- Capabilities
  - Type of materials
  - Gases
  - Temperatures

## Materials
- Chemical composition
- Sample preparation

## SOP
- Test method
- Repetitions
- Number of samples

## Results
- Number of repetitions
- Number of samples
- Average
- Uncertainty

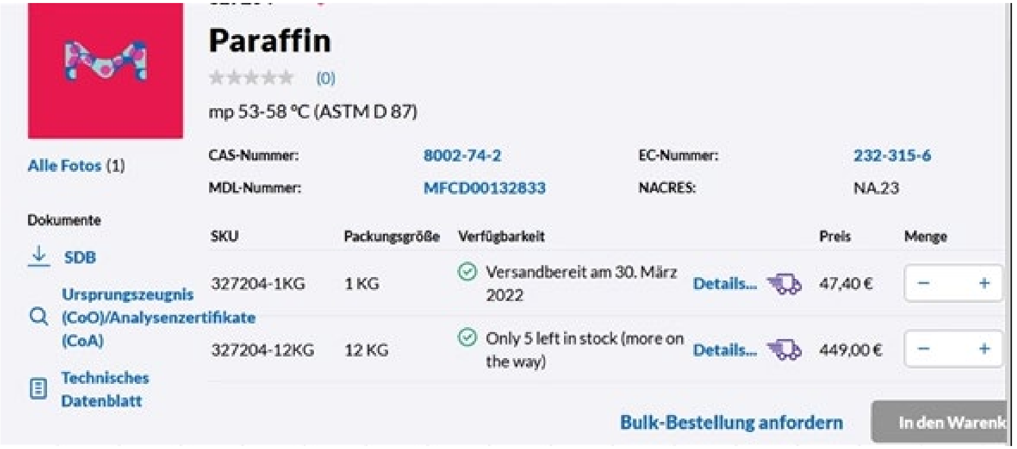
## Analysis and statistics
- Data analysis
- Discussion and interpretation

# RRT-Data Collection

## Measurement capabilities for Thermal Conductivity/Thermal Diffusivity

- Main specifications for the test procedure:
- Sample form/size
  - LFA solid **12.7 mm** ϕ
  - THW **50 ml**;
  - TPS **18 mm** ϕ; 6-30 mm ϕ; 4-29 mm ϕ; 2-29 mm ϕ
  - THB **25 x 25x 5 mm**; 10x20x3 mm; 20x22x3; 100 mL
- Temperature
  - LFA RT to 500 °C (-120 to 2800 °C);
  - THW RT to150 °C ( -40 to180 °C)
  - TPS RT to 180 °C (-70 to 300 °C)
  - THB -15 to 200 °C ( -150 to 700 °C)
- Atmosphere
  - Air/ protective gas
  - Lab conditions
  - Less available
    - Argon
    - Vacuum

# RRT-Material



Paraffin

(0)

mp 53-58 °C (ASTM D 87)

Alle Fotos (1)

| | | | |
|---|---|---|---|
| CAS-Nummer: | 8002-74-2 | EC-Nummer: | 232-315-6 |
| MDL-Nummer: | MFCD00132833 | NACRES: | NA.23 |

Dokumente

SDB

Ursprungszeugnis (CoO)/Analysenzertifikate (CoA)

Technisches Datenblatt

| SKU | Packungsgröße | Verfügbarkeit | | Preis | Menge |
|---|---|---|---|---|---|
| 327204-1KG | 1 KG | Versandbereit am 30. März 2022 | Details... | 47,40 € | − + |
| 327204-12KG | 12 KG | Only 5 left in stock (more on the way) | Details... | 449,00 € | − + |

Bulk-Bestellung anfordern

In den Warenk

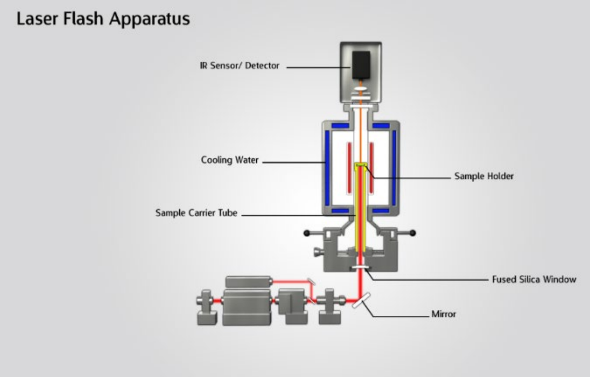
Paraffin CAS No. 8002-74-2, Product No. 327204

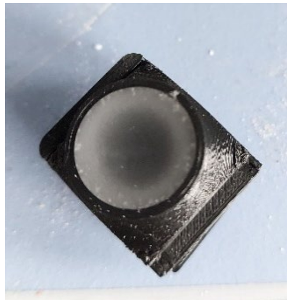
## Sample preparation

- Two set of samples with two different cooling rates
  - Heating up to 70 °C ( till liquid state) and then cool down at Tamb
  - Heating up to 70 °C and then place it in an oven at 50 °C for solidification.
- Check density, bubbles
- Heat up till fully melted (transparent)-participants add time in each case

# RRT-Procedures

- Focus the laser to maximal 6 mm (non focusable LFA: use an aperture with max. 6 mm) to avoid signal superposition with the PTFA ring.
- Use thickness values from the additional information for the thermal diffusivity measurements.
- Perform measurements with at least 5 shots at 25 °C, 40 °C and 50 °C with
  - the pulse length and voltage recommended by the software or recommended for polymers by the manufacturer.
  - ca. 50 % pulse energy. It is recommended to adjust pulse energy by changing the pulse length.
  - ca. 75 % pulse energy.
  - ca. 125 % pulse energy.
- Provide information about
  - Pulse voltage
  - Pulse length
  - Pulse area (integral of pulse voltage)
  - Used evaluation model

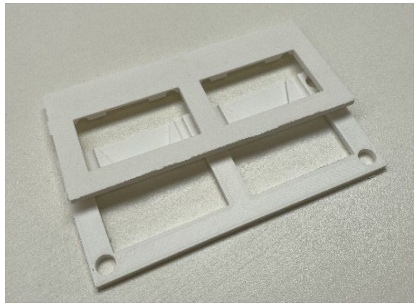
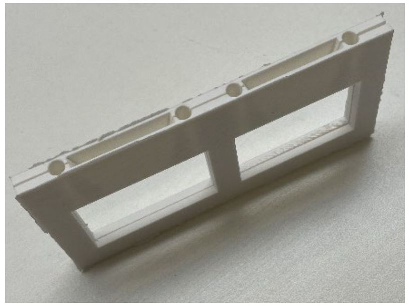
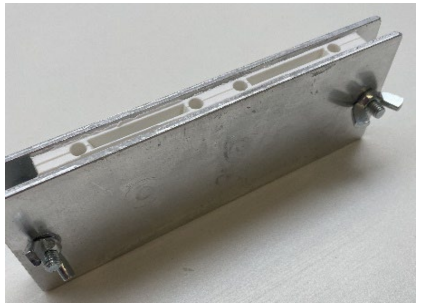


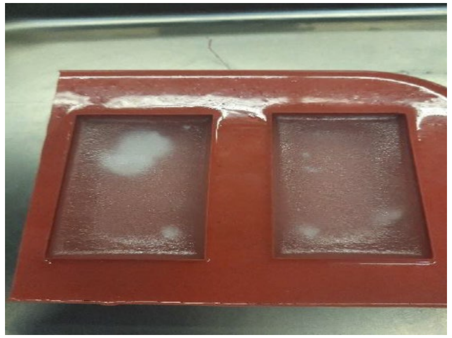



# RRT-Procedures

## Solid Samples

- Two sample halves with flat and plane-parallel to the sensor surface get good thermal contact with the sensor.
- Samples preparation steps (optional)
- 3D printed moulds between aluminium plates (volume for expansions in 3D printed moulds to avoid pores formation).

- Melt the PCM
- Heat the moulds
- Fill/Inject the PCM
- Cooling at Tamb and cool at 50 °C
- Extract the sample “carefully.”
- Check thickness (ideally ± 0.05mm)

# RRT-Data Analysis

The standard uncertainty is the standard deviation of the mean:

$$u(\bar{q}) = s(\bar{q}) = \frac{s(q_k)}{\sqrt{n}}$$

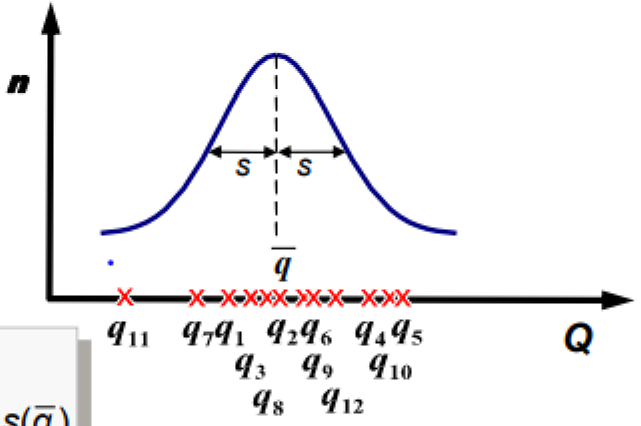
$$u(\bar{q}) = s(\bar{q}) = \sqrt{\frac{1}{n \cdot (n-1)} \sum_{j=1}^{n} \left(q_j - \bar{q}\right)^2}$$

$n$ number of observations

$q_j$ result of observations $j$

$\bar{q}$ mean of the $n$ observations

$s(\bar{q})$ standard deviation of the mean




**GUM 4.2.3:**

The **experimental standard deviation of the mean** $s(\bar{q})$ quantifies how well $\bar{q}$ estimates the expectation of $q$, and may be used as a **measure of the uncertainty of** $\bar{q}$ .

Main steps to the determination of measurement uncertainty according to GUM (Version 8/2015) Authors: W.Schmid (EURAMET), S.Mieke (PTB), M.Hoxha (DPM) Copyright © EURAMET e.V. 2015

# RRT-Data Analysis

Expanded uncertainty: $U = k \cdot u_c$

Gaussian or normal distribution:

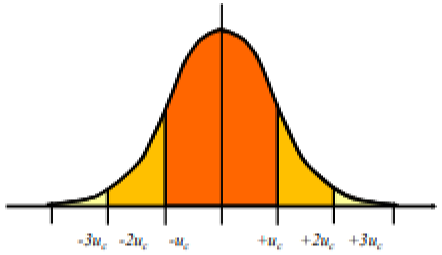



Table G.1 — Value of the coverage factor $k_p$ that produces an interval having level of confidence $p$ assuming a normal distribution

| Level of confidence $p$ (percent) | Coverage factor $k_p$ |
|---|---|
| 68,27 | 1 |
| 90 | 1,645 |
| 95 | 1,960 |
| 95,45 | 2 |
| 99 | 2,576 |
| 99,73 | 3 |

Measurement result:

$Y = y \pm U$ with $k$

Y Measurand

y Best estimate of the measurand

U Expanded uncertainty







*Main steps to the determination of measurement uncertainty according to GUM (Version 8/2015) Authors: W.Schmid (EURAMET), S.Mieke (PTB), M.Hoxha (DPM) Copyright © EURAMET e.V. 2015*

# Challenges

- Equipment
  - Different equipment based in different methods
    - Transient methods
    - Steady state methods
- Sample
  - Different sample size
    - Different sample preparation
- Results
  - Uncertainty contribution
    - Systematic uncertainty
    - Experimental uncertainty

# Next Steps

- Analyse all the values measured and assess uncertainties
- Compare uncertainties
  - similar techniques
  - Different techniques
- Start optimized RRT round
- Explore new materials
  - PCM (liquid state, inorganic)
  - TCM

# Thank you for your attention

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