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**International Energy and Environment Conference (IEEC) 2025**

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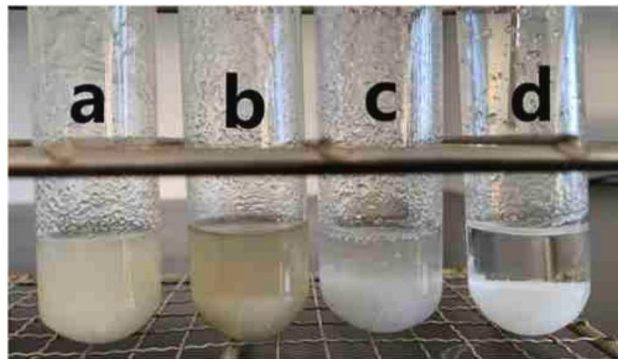
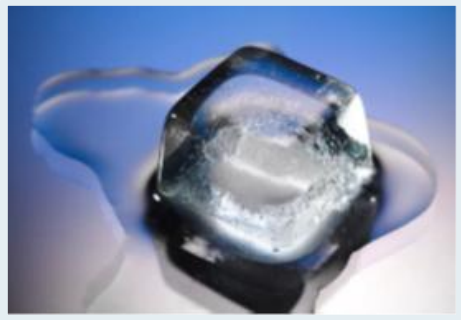
# Calorimetry of Phase Change Materials (PCM) for Heat Storage Applications

# 1. Motivation

## Heat Storage with Phase Change Materials (PCM)

Latent heat storage:  $dQ = \Delta h_{melt} * dm_{s \rightarrow l}$

- Salt hydrates as PCM<sup>1</sup>:
  - In general cheap and available
  - High heat storage density
  - Not flammable
  - **Likely to supercool**
  - **Phase segregation**



Phase segregation in salt hydrate PCM with varying gelling agent content.<sup>2</sup>

RAL Quality Mark: RAL-GZ 896 – Phase Change Materials

- Measurement (Salt Hydrates)  $T_m$  and  $\Delta h_{fl}$ :
  - T-history Calorimeter
  - Large Scale DSC
  - 3 Layer Calorimeter
    - Sample containment can be used for thermal cycling
- 3 Layer Calorimeter not well documented in literature (only gray literature<sup>3</sup>):



<sup>1</sup>Stephan, Peter (Hg.) (2019): VDI-Wärmeatlas. Fachlicher Träger VDI-Gesellschaft Verfahrenstechnik und Chemieingenieurwesen. Springer-Verlag GmbH. 12. Auflage 2019. Berlin: Springer Vieweg (Springer Reference Technik).

<sup>2</sup>Gao, Liqiang; Zhang, Xuelai; Hua, Weisan (2024): Preparation and thermal properties of sodium carbonate decahydrate as a novel phase change material for energy storage. In: Journal of Energy Storage 84, S. 110723. DOI: 10.1016/j.est.2024.110723.

<sup>3</sup>Laube, Andreas: The 3-layer calorimeter. Hg. v. Andreas Laube. w & a - wärme- und anwendungstechnische Prüfungen Andreas Laube. Online verfügbar unter <http://wunda.tech/3-Schicht-Kalorimeter/>, zuletzt geprüft am 14.05.2025.

# Agenda

## Investigation of the 3-layer calorimeter

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1. Motivation
2. Introduction to the 3-layer calorimeter
3. Fraunhofer ICT version of the 3-layer calorimeter
  - a) **Construction**
  - b) **Testing**
  - c) **Modeling**
  - d) **Calibration**
4. Conclusion

## 2. Introduction to the 3-layer calorimeter

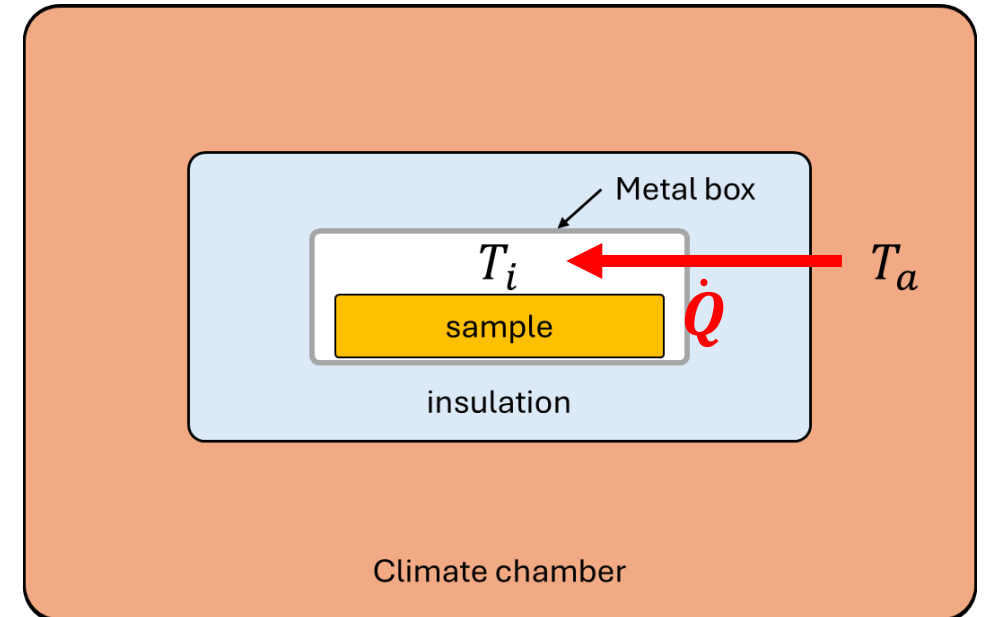
### Assumptions:

- Temperature inside calorimeter  $T_i$  is homogenous
- Temperature around calorimeter  $T_a$  is homogenous
- Thermal resistance of calorimeter  $R_{cal}$  is known
- Thermal capacity of calorimeter  $C_{p,cal}(T_i)$  is known
- Sample temperature equals internal temperature of calorimeter  
 $T_{sample} = T_i$

} **true?**  
} **how?**

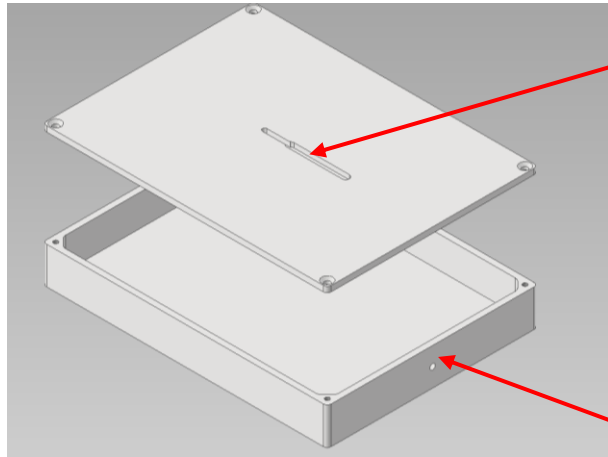
### Measurement principle:

- Heat flow  $\dot{Q}$  at  $T_i$  can be calculated from recorded temperatures
- Total thermal capacity  $C_{p,total}(T_i)$  can be calculated from  $\dot{Q}$  and  $\dot{T}_i$
- Thermal capacity of sample  $C_{p,sample}(T_i) = C_{p,total}(T_i) - C_{p,cal}(T_i)$



# Fraunhofer ICT version of the 3-layer calorimeter

## a) Construction



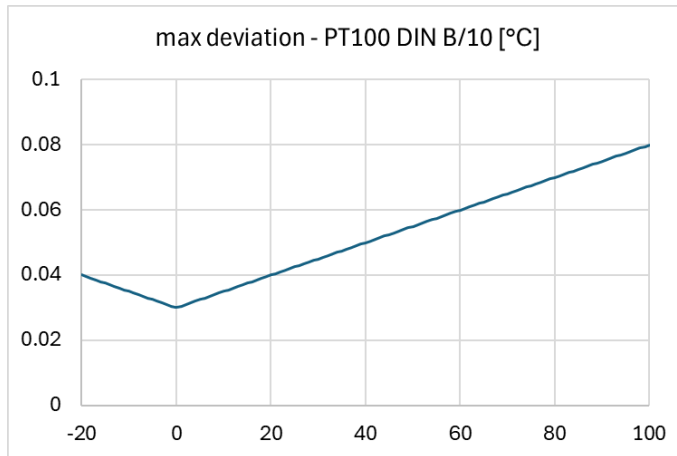
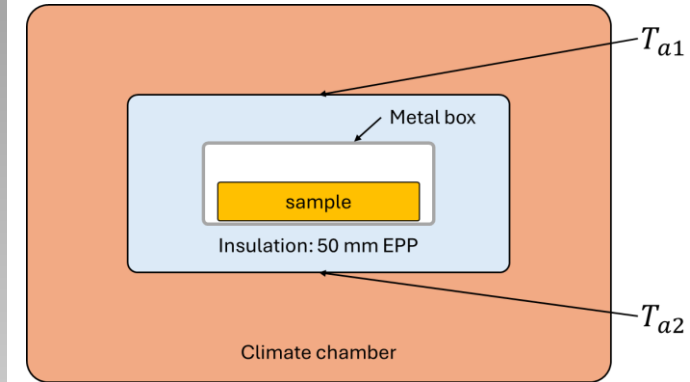
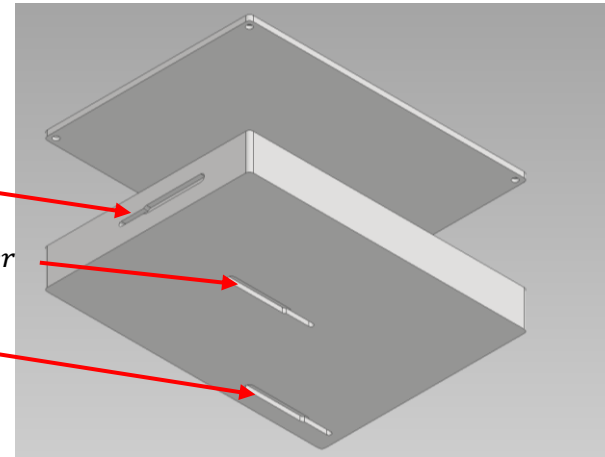
inside top:  $T_{i,1}$

inside side:  $T_{i,2}$

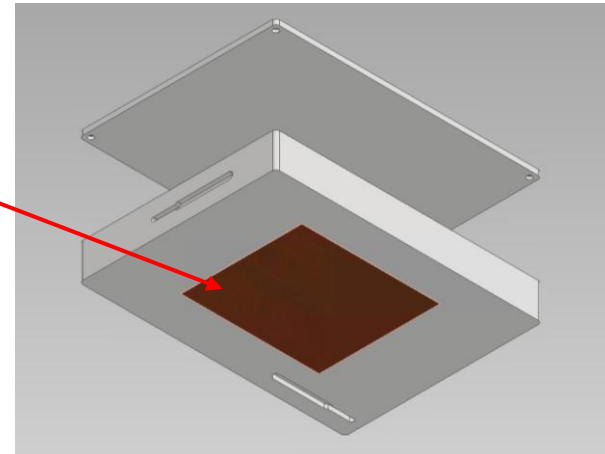
inside bottom (heater):  $T_{i,heater}$

inside bottom corner:  $T_{i,3}$

inside sample:  $T_{i,sample}$



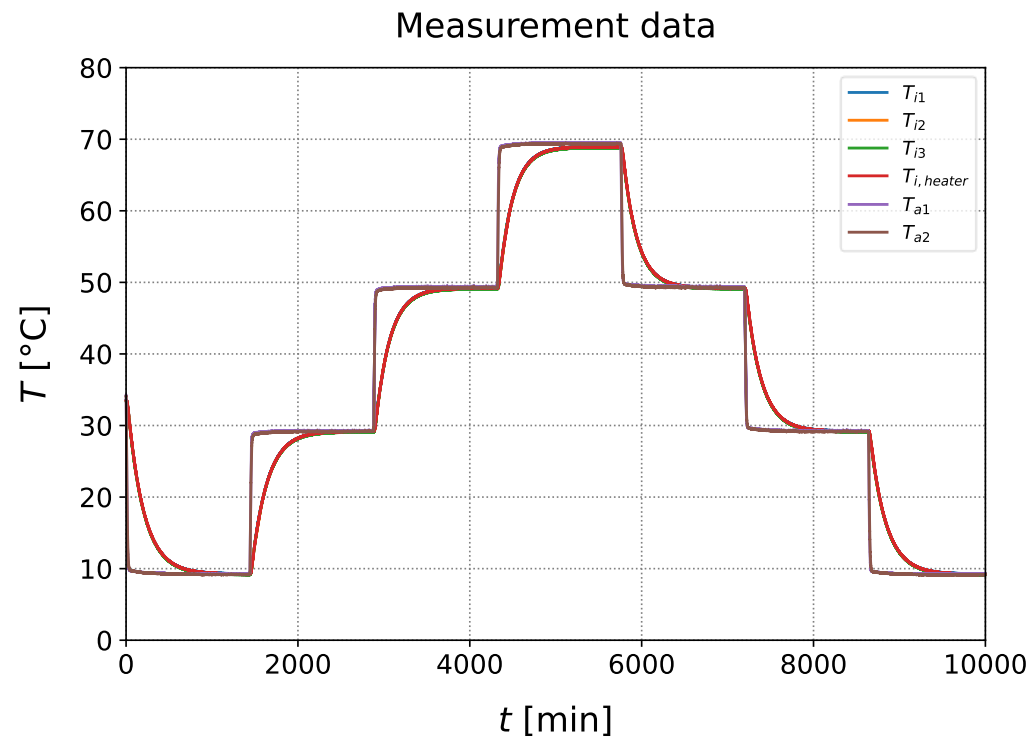
Foil heater



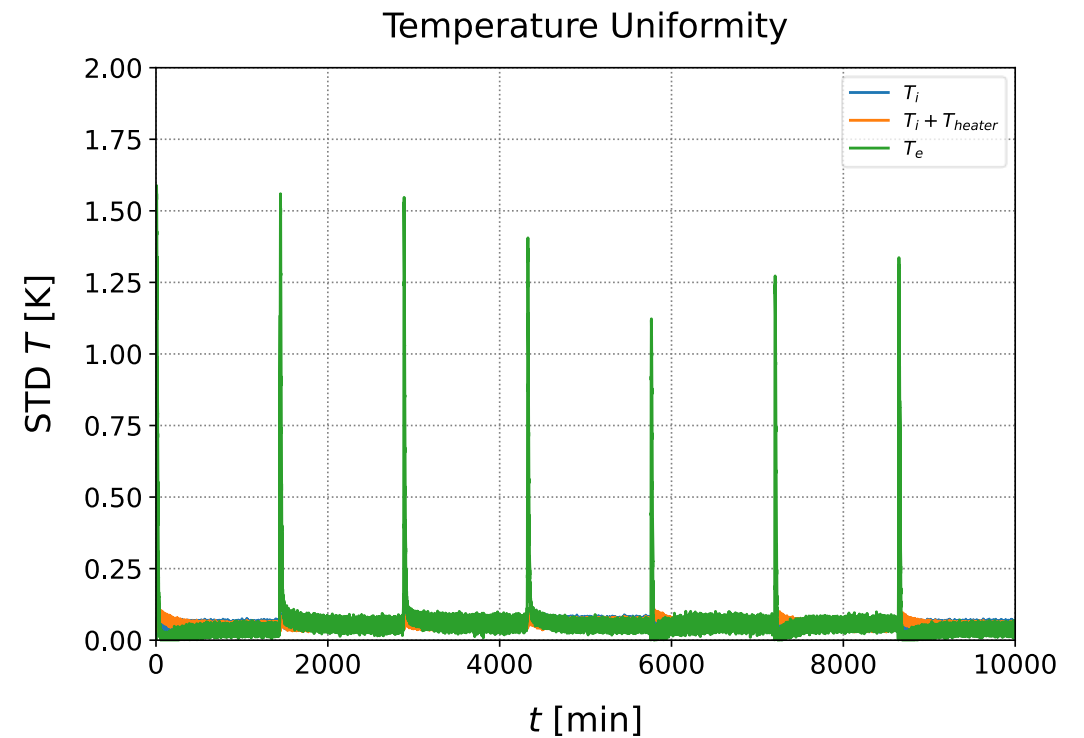
# Fraunhofer ICT version of the 3-layer calorimeter

## b) Testing – heating / cooling without phase transition (sample = water, 111 g)

### Temperature profiles



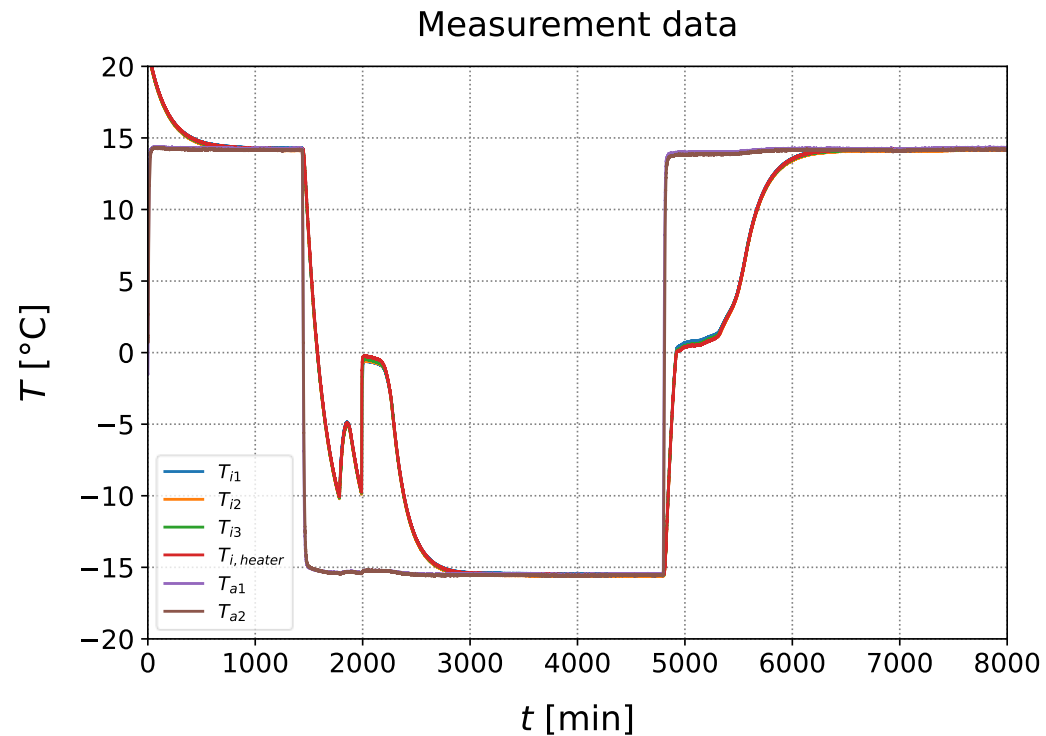
### Temperature deviations inside and outside the calorimeter



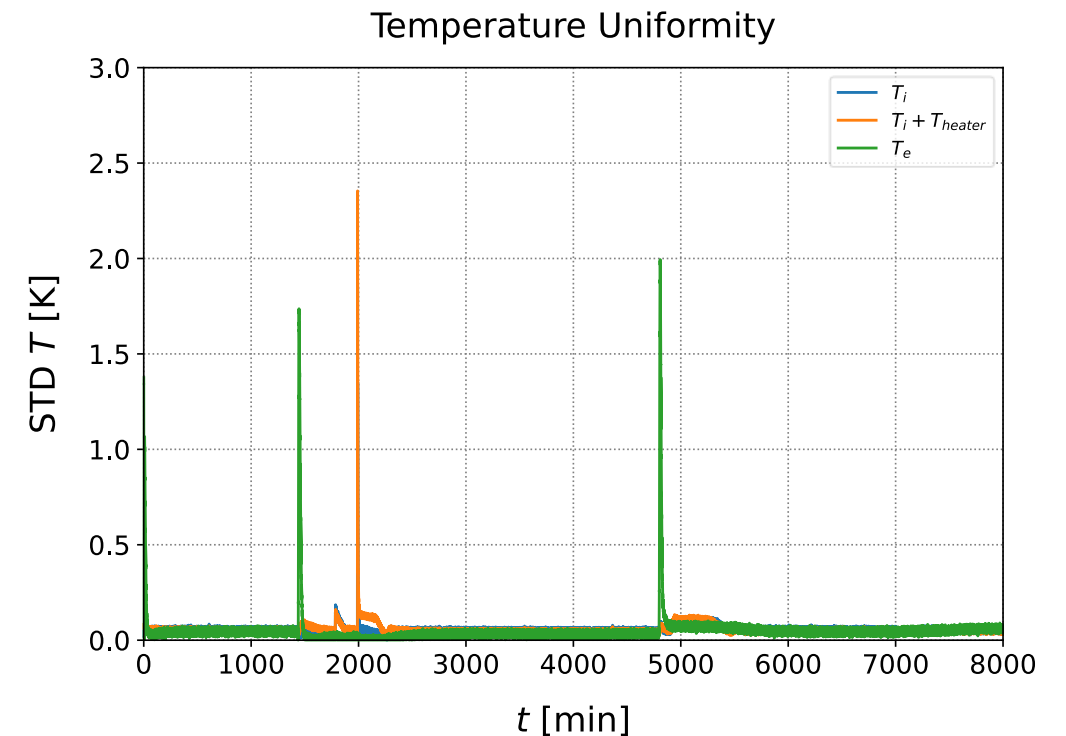
# Fraunhofer ICT version of the 3-layer calorimeter

## b) Testing – heating / cooling with phase transition (sample = water, 111 g)

### Temperatures



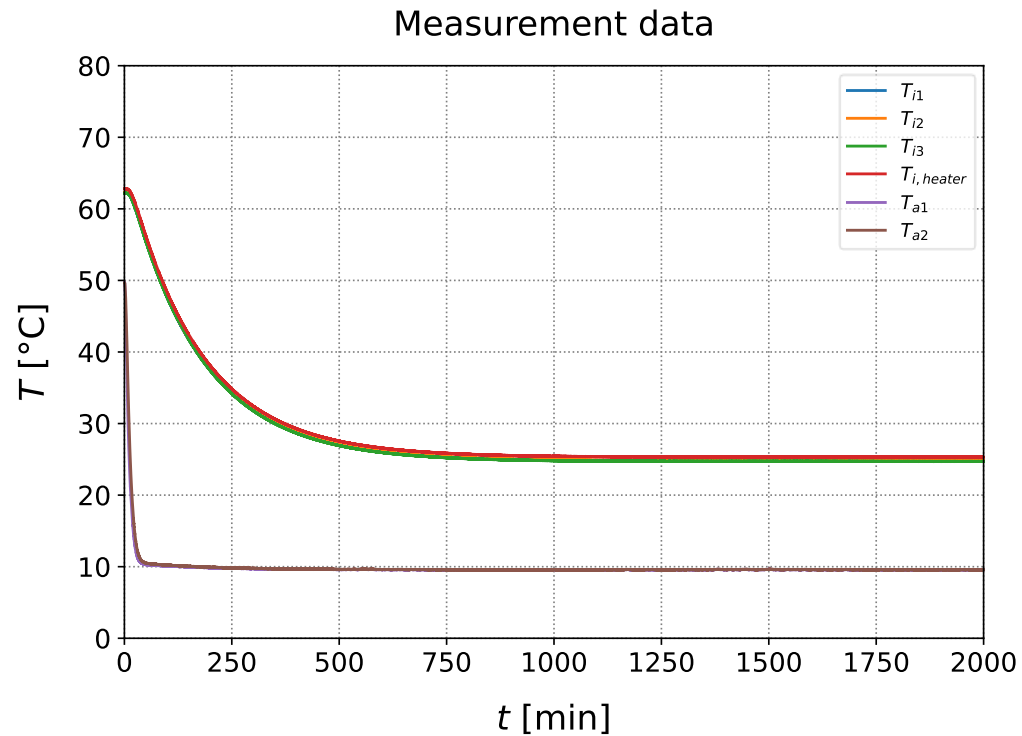
### Temperature deviations inside and outside calorimeter



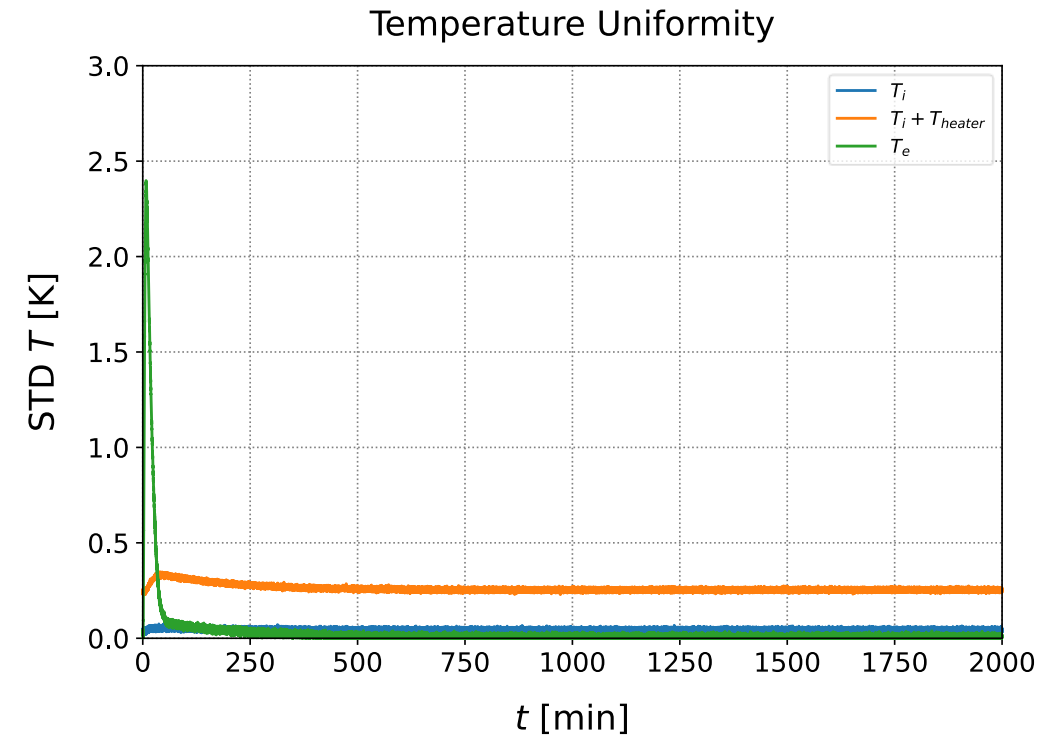
# Fraunhofer ICT version of the 3-layer calorimeter

b) Testing – heating / cooling without phase transition with heater (**P = 1 W, sample = water, 111 g**)

## Temperature profiles



## Temperature deviations inside and outside the calorimeter



# Fraunhofer ICT version of 3 layer calorimeter

## c) Modeling

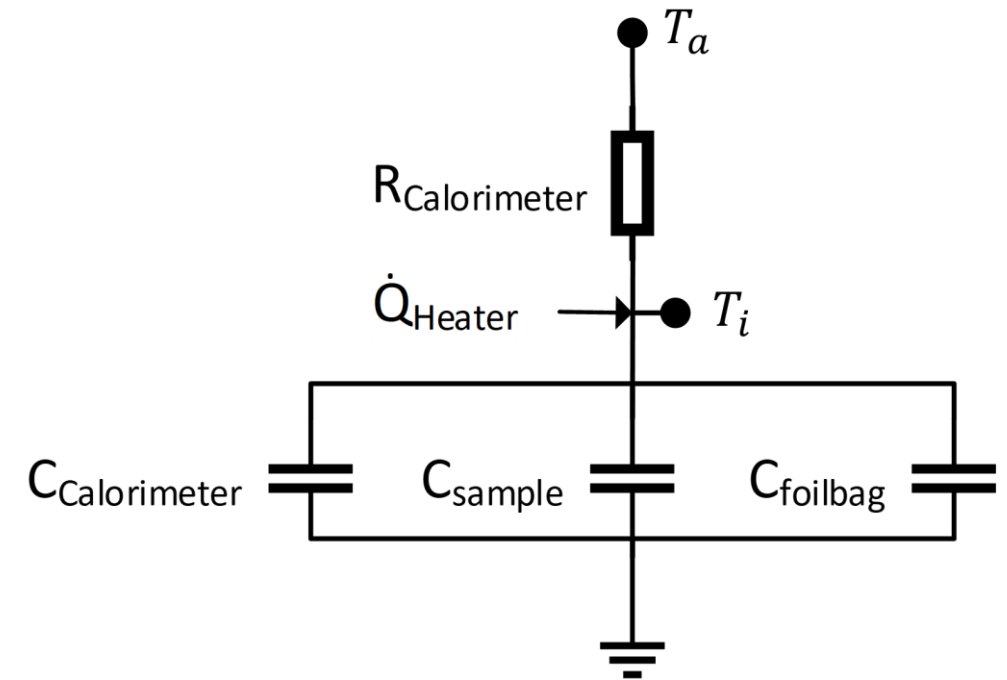
3 layer calorimeter is modeled as thermal node model:

$$\dot{T}_i = \left( \frac{T_a - T_i}{R_{cal}(T)} + \dot{Q}_{heater} \right) \cdot \frac{1}{C_{sample}(T) + C_{cal}(T) + C_{foilbag}(T)}$$

**Stationary  $\dot{T}_i = 0$ :**

$$\frac{T_a - T_i}{R_{cal}(T)} + \dot{Q}_{heater} = 0$$

$$R_{cal}(T) = \frac{T_i - T_a}{\dot{Q}_{heater}}$$

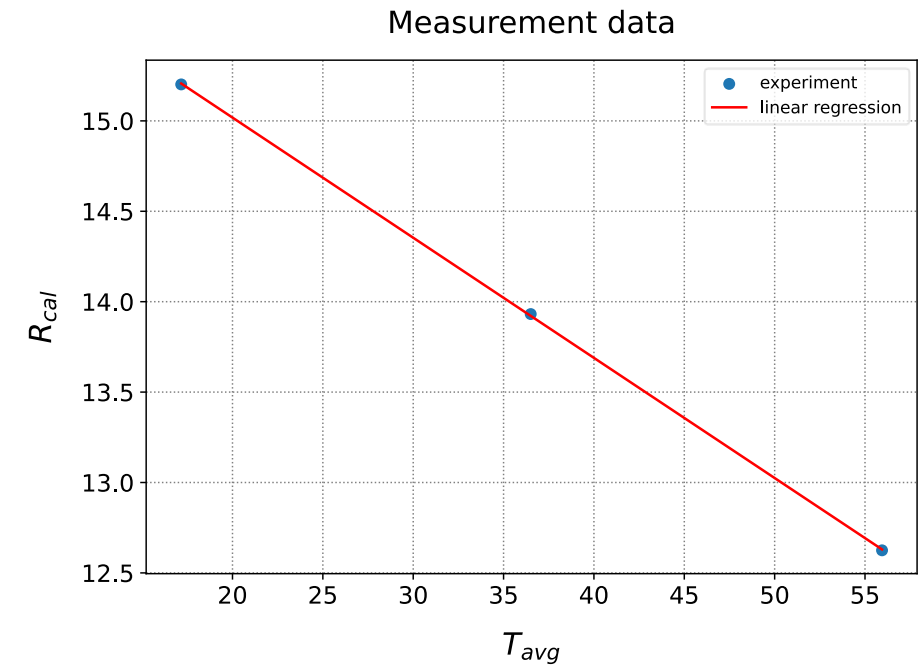
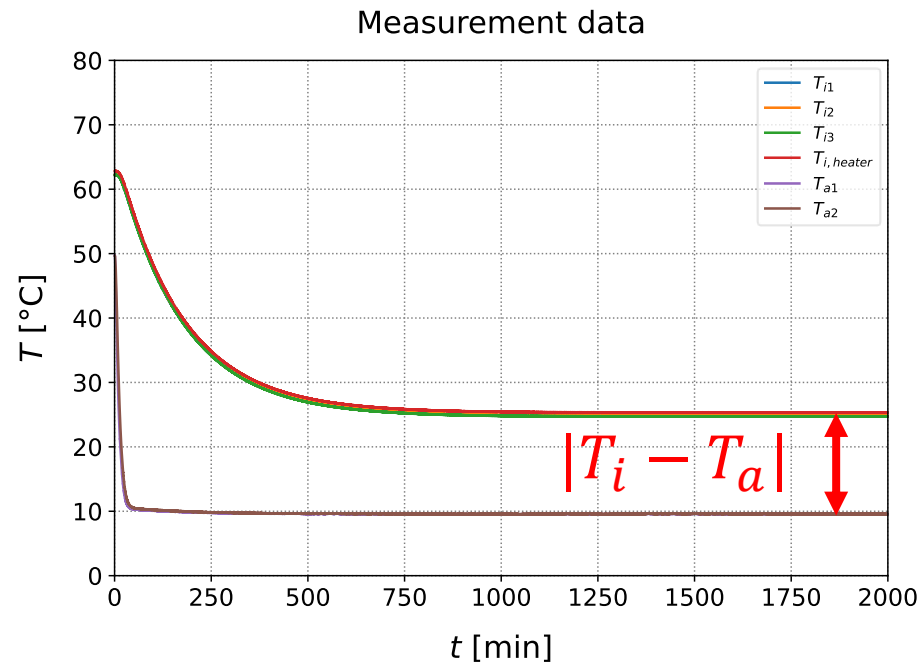


# Fraunhofer ICT version of 3 layer calorimeter

## d) Calibration – Thermal Resistance

### Thermal Resistance

$$R_{cal} \left( \frac{T_i + T_a}{2} \right) = \frac{T_i - T_a}{\dot{Q}_{heater}}$$

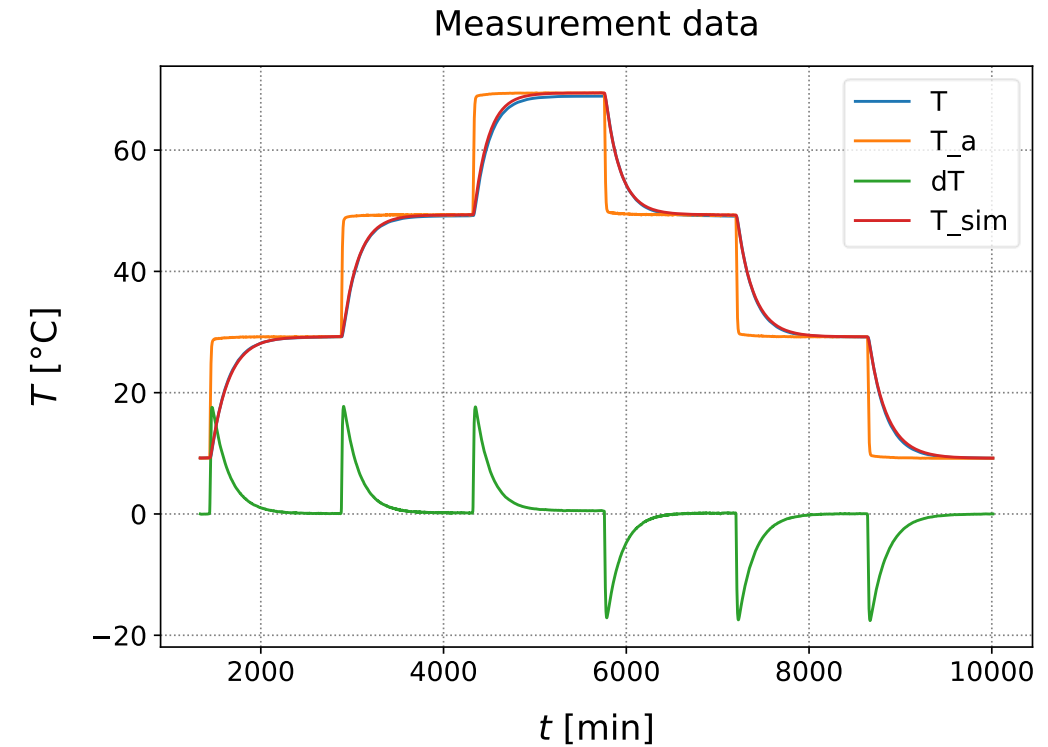
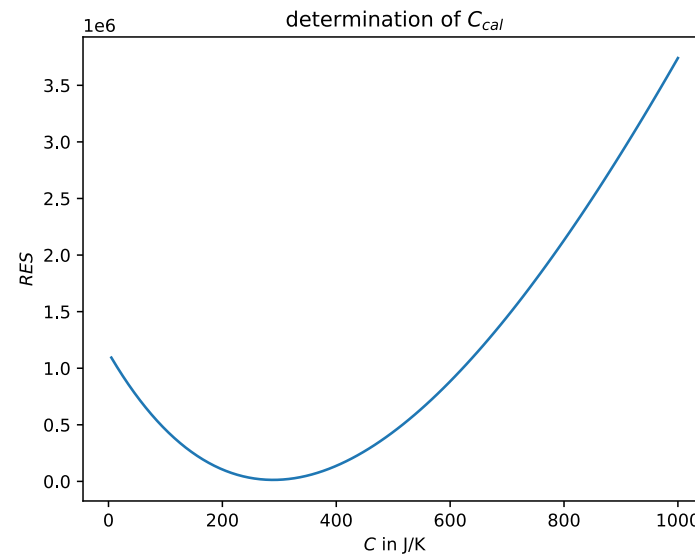
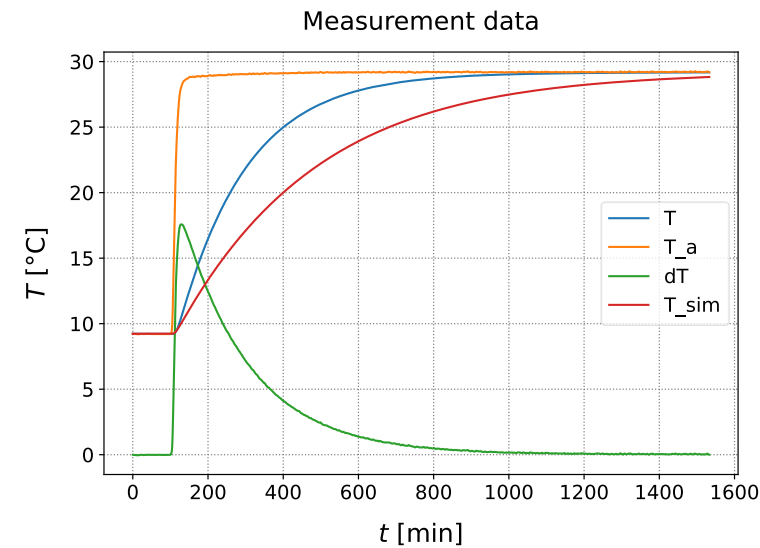


# Fraunhofer ICT version of 3 layer calorimeter

## d) Calibration – Thermal Capacity

### Procedure:

- Differential equation for the calorimeter is solved numerically.
- Deviation from simulation results is minimized by adjusting  $C_{cal}$ .
- All measurements without phase change are used in this procedure.
- Thermal capacity determined as 289.2 J/K.



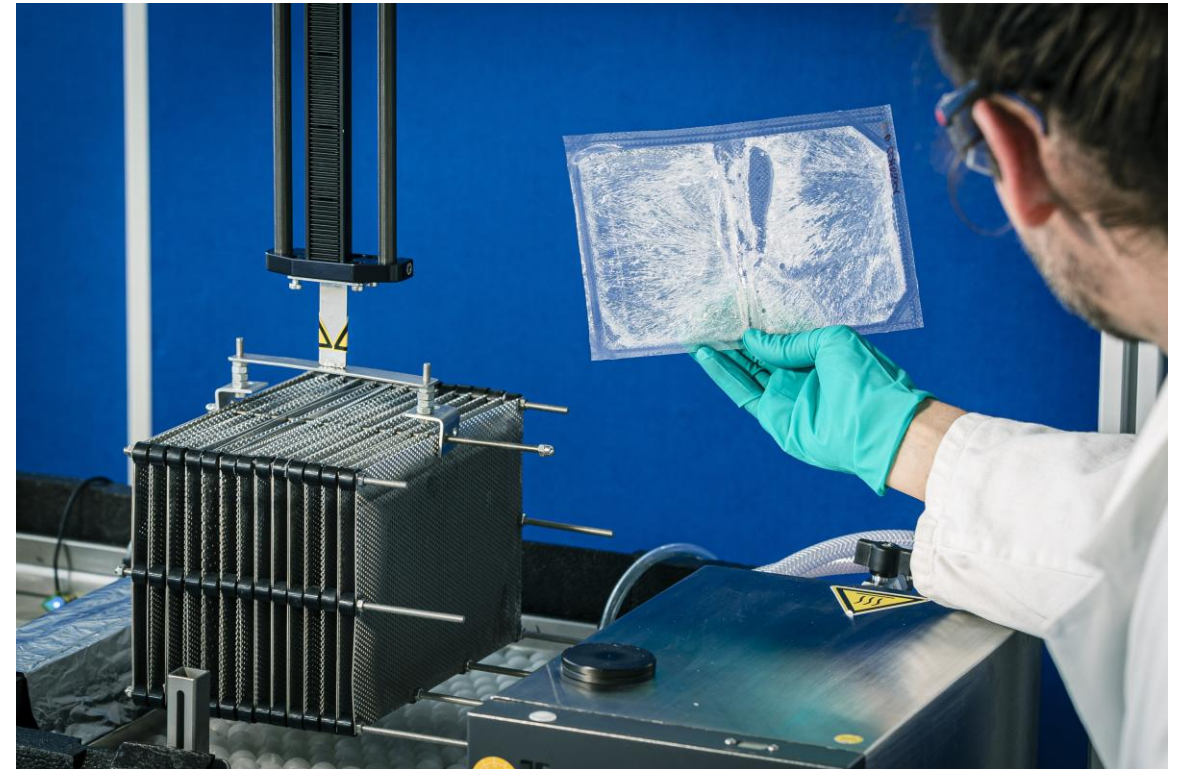
# Results and Outlook

## Results:

- Temperature uniformity was demonstrated
- Electrical heater was implemented for calibration
- A two-step calibration procedure was developed
- The 3-layer calorimeter is suitable for thermal cycling investigations of salt hydrate-based PCM

## Ongoing Work:

- Statistical analysis of measurement uncertainty
- Validation with reference PCM
- Improving accuracy using advanced modeling methods
- Reducing the number of sensors



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