# Do differences in bench or small scale experiments manifest in different fire growth behaviour? A case study for PMMA

10/18/2022 I Karen De Lannoye



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# Introduction

- Model validation
  - Small scale: input for modelling
  - Large scale: validation of models
  - E.g. Christifire [1],[2]
- Comparison of material for reconstructive fire testing [3]
  - Similarity of material based on MCC or cone
- Assessing fire hazard from small-or bench scale data (e.g. [4,5])
- Comparison between black cast and transparent extruded PMMA [6]

# Introduction

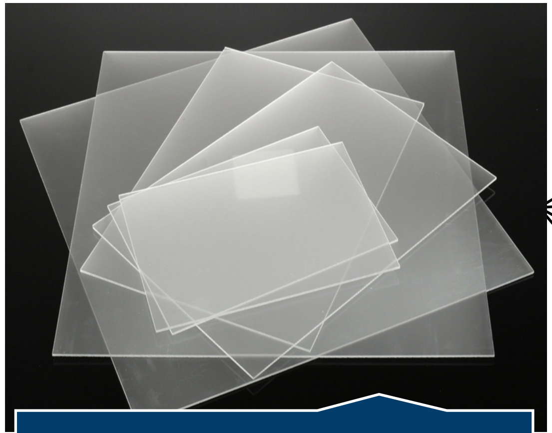

Fig: [7]

PMMA samples

TGA → Kinetic parameters

MCC → Heat of combustion

Cone calorimeter → Thermophysical parameters

Gasification apparatus → Thermophysical parameters

Identify differences in material parameters

Larger scale flame spread

# Do differences in milligram scale experiments manifest in different behaviour in bench scale experiment ? A case study for $CaCO_3$

10/18/2022 I Karen De Lannoye



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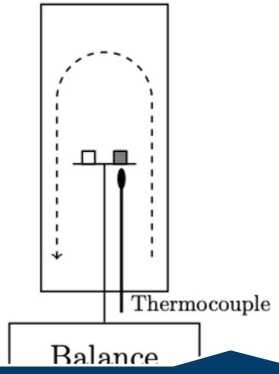
# Introduction

$CaCO_3 \rightarrow CaO + CO_2$

CaCO3




TGA

Tube furnace

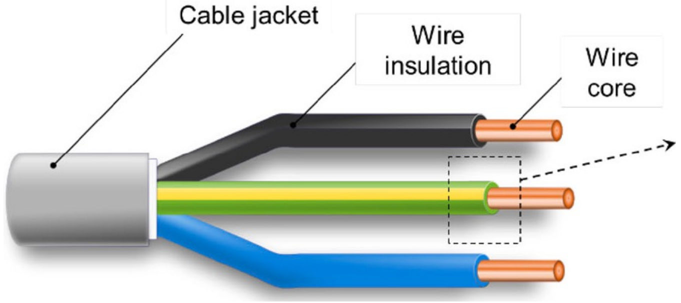
# Introduction

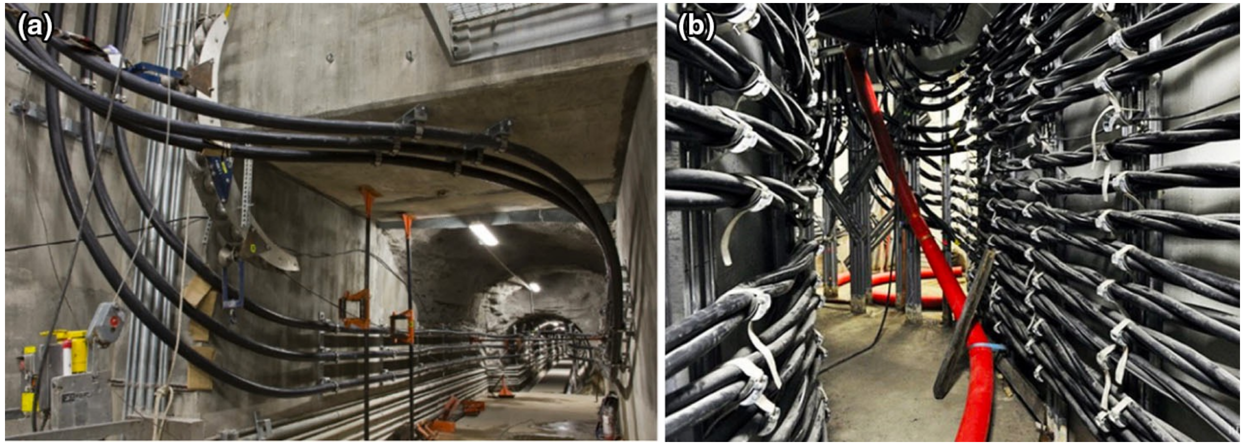
➢ Objective of thesis: Study the pyrolysis of cable fires

- Potential source of fire: residential buildings, nuclear power plants, aircrafts, spacecrafts,…
- Complex combined system of metal core and insulation

- Gap between experimental data and modeling
  - Different boundary conditions

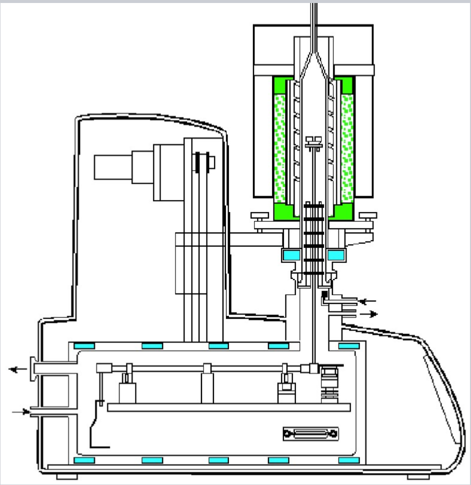
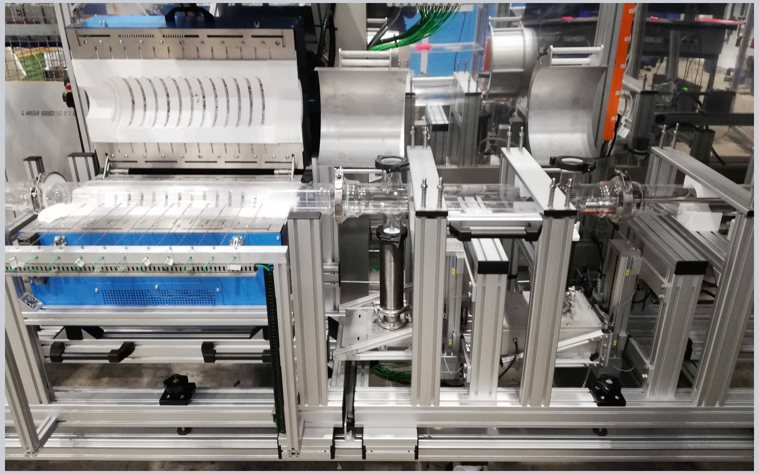
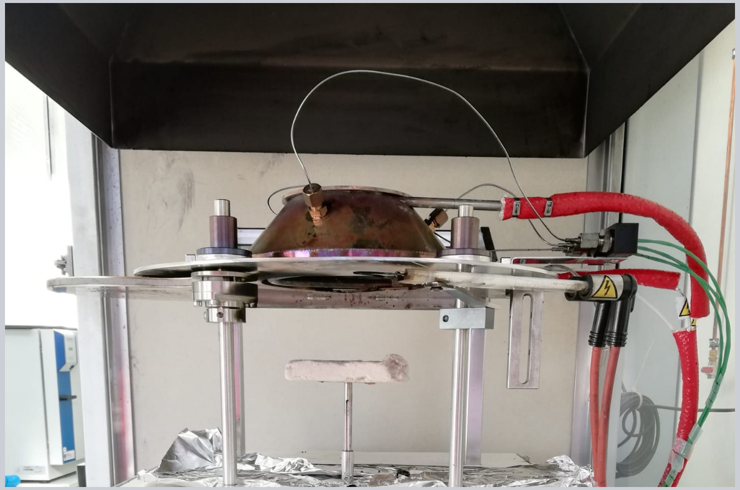
➢ Experiments with well-known boundary conditions to improve modelling

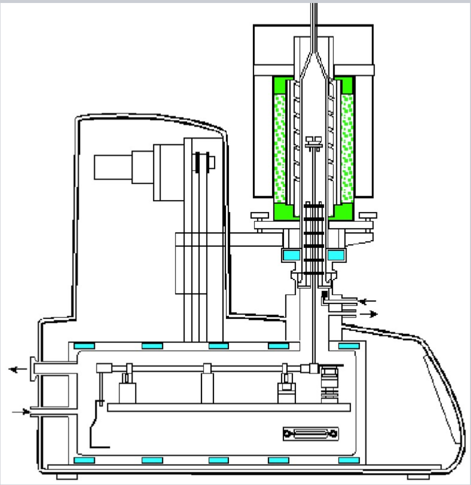
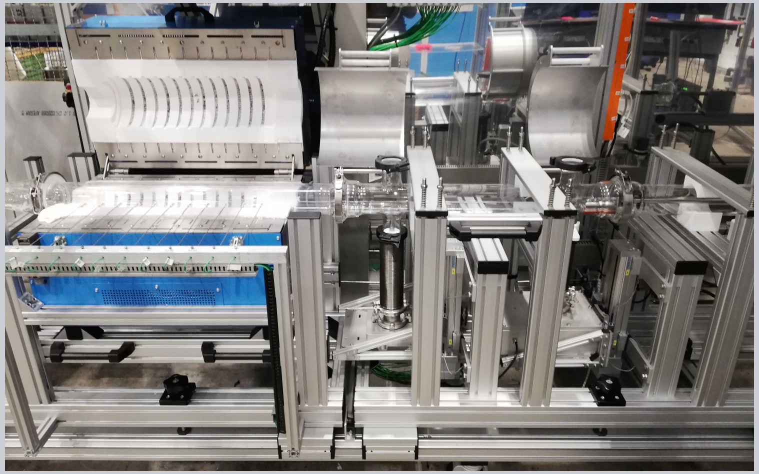
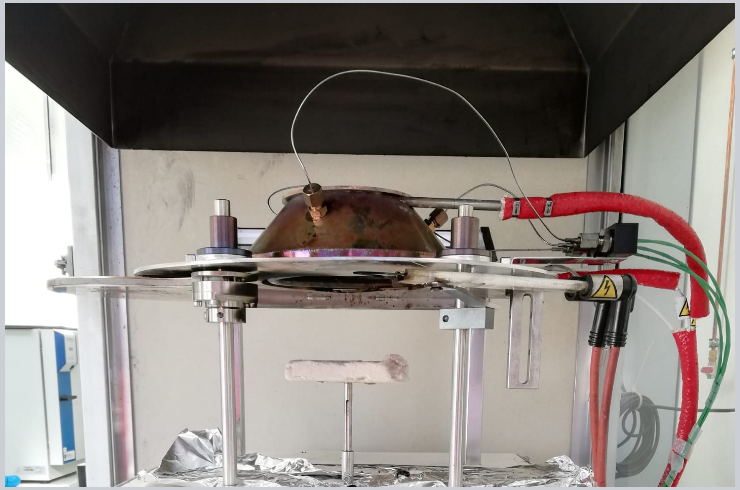




Ref. fig: [8]

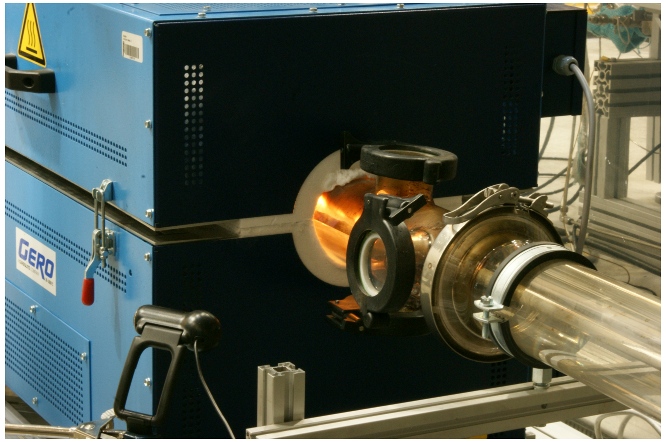
# Introduction

| Thermogravimetric analyser | Tube furnace | Cone calorimeter |
|---|---|---|
| Sample size: mg<br><br>Ref. fig: [9] | Sample size: 60 cm<br> | Sample size: 10 cm x 10 cm<br> |
| • Heating rate<br>• Radial symmetric heating | • Heating rate of temperature<br>• Radial symmetric heating | • Heat flux<br>• Top heating |

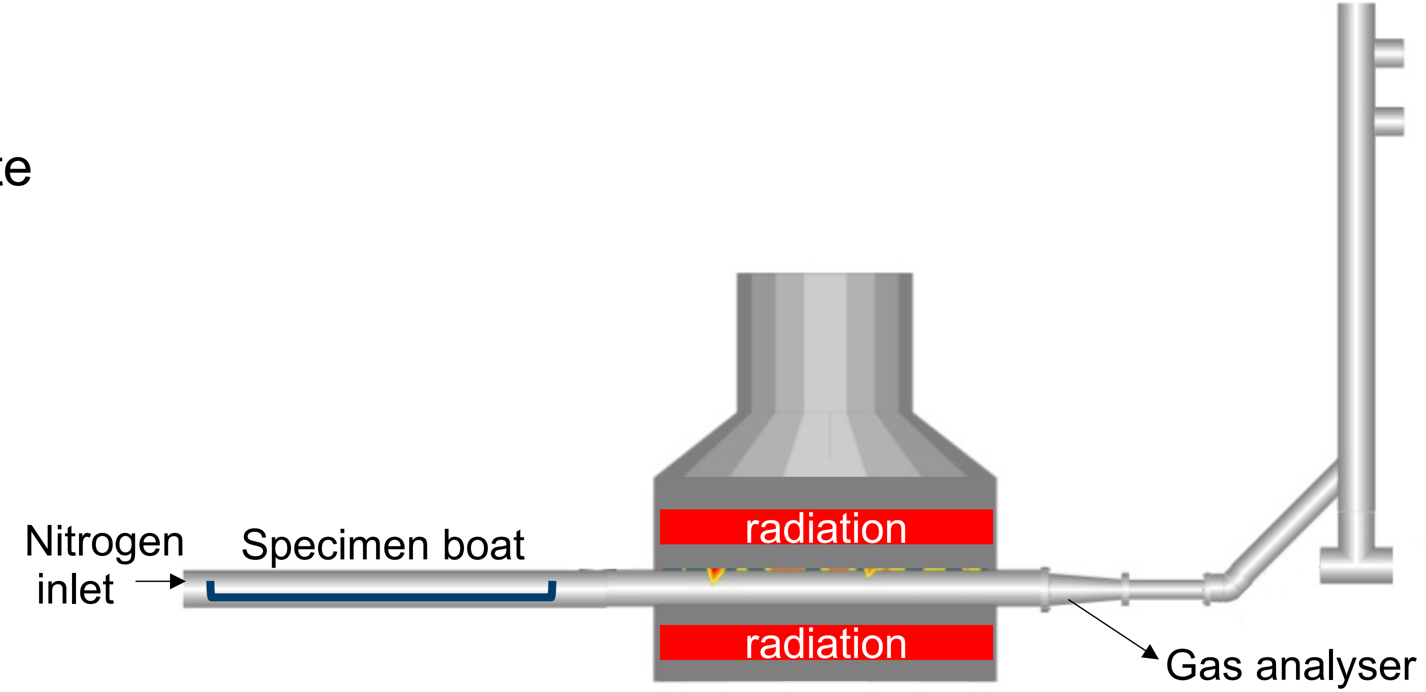
# Introduction

| Thermogravimetric analyser | Tube furnace | Cone calorimeter |
|---|---|---|
| Sample size: mg<br><br>Ref. fig: [9] | Sample size: 60 cm<br> | Sample size: 10 cm x 10 cm<br> |
| + Well controlled boundary conditions<br><br>Amount of material<br>Lack of heat feedback | + Well controlled boundary conditions<br>+ Representative amount of sample material | + Representative amount of sample material<br><br>Open → boundary conditions not controlled |

# Tube furnace




- Specimen size: 60 cm to 80 cm (by 5 cm by 2 cm)
- Inner diameter: 10 cm
- Movable specimen boat
  - Experiments at specific temperature
  - Experiments with a certain heating rate
- Maximal temperature: 1000˚C
- Maximal heating rate: 5 ˚C/min
- Analytics: CO, $CO_2$,$O_2$
  → Heat release rate
- Controlled atmosphere



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# Tube furnace

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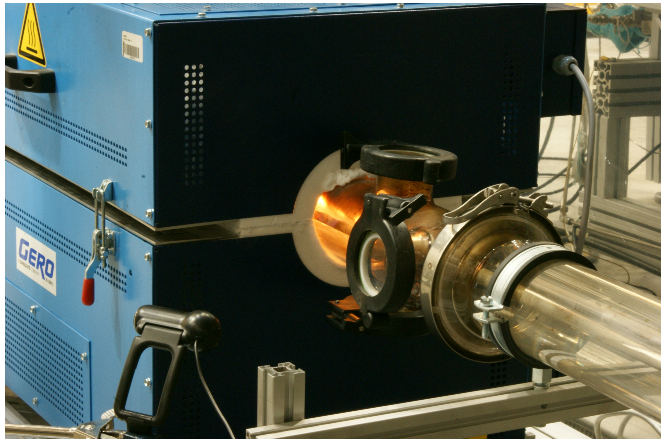
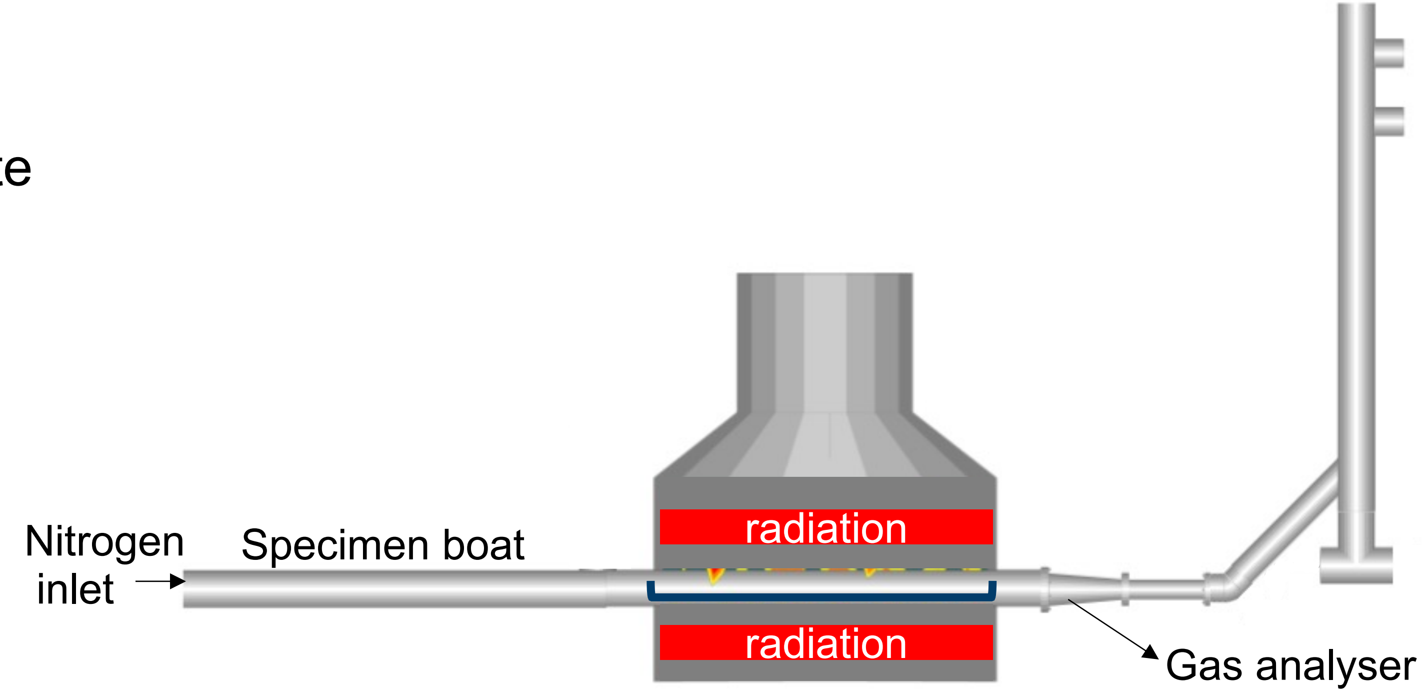




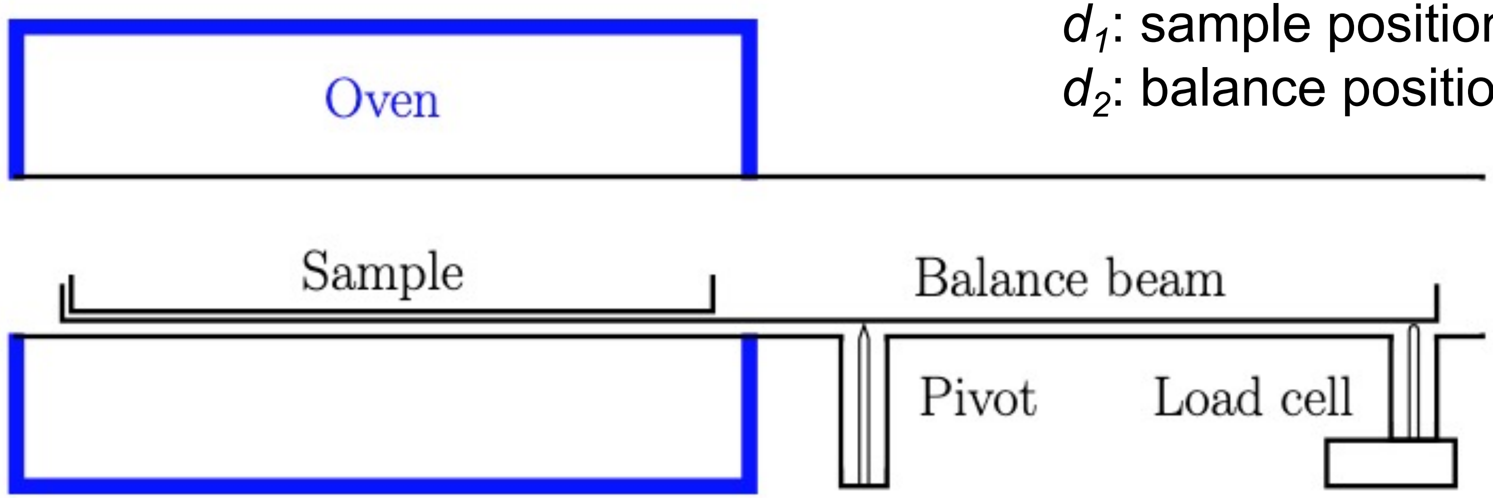
# Tube furnace

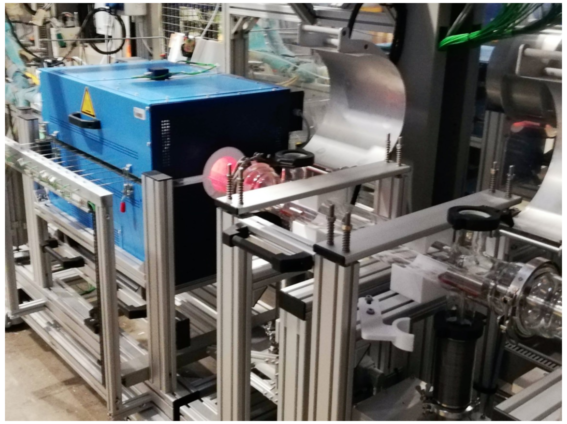
- Both for isothermal as for dynamic experiments
- Cantilever from quartz glass:
  sample on one side, weighing cell on the other side

$$F_{balance} = \frac{m_1 g\, d_1}{d_2}$$

- Validation with reference weights

$F_{balance}$: force on the balance
$g$: gravitational constant
$m_1$: sample mass
$d_1$: sample position
$d_2$: balance position





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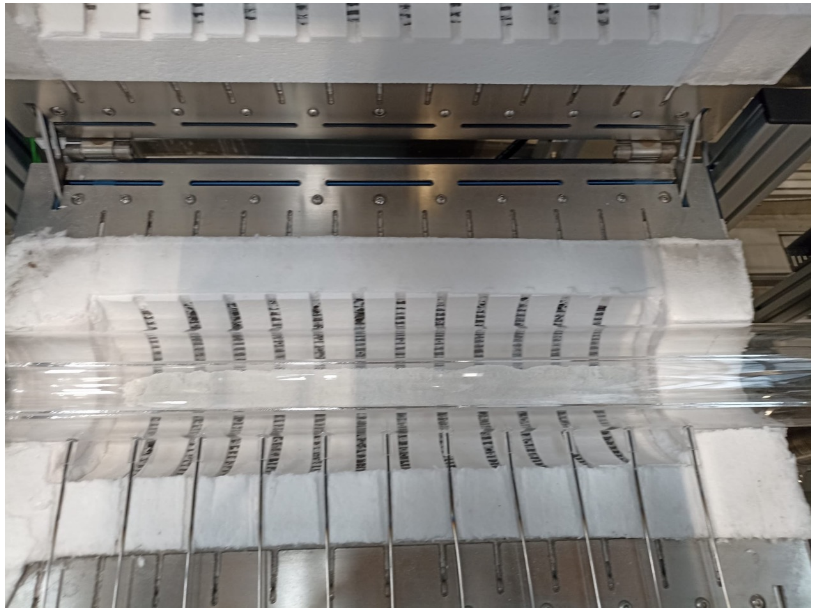
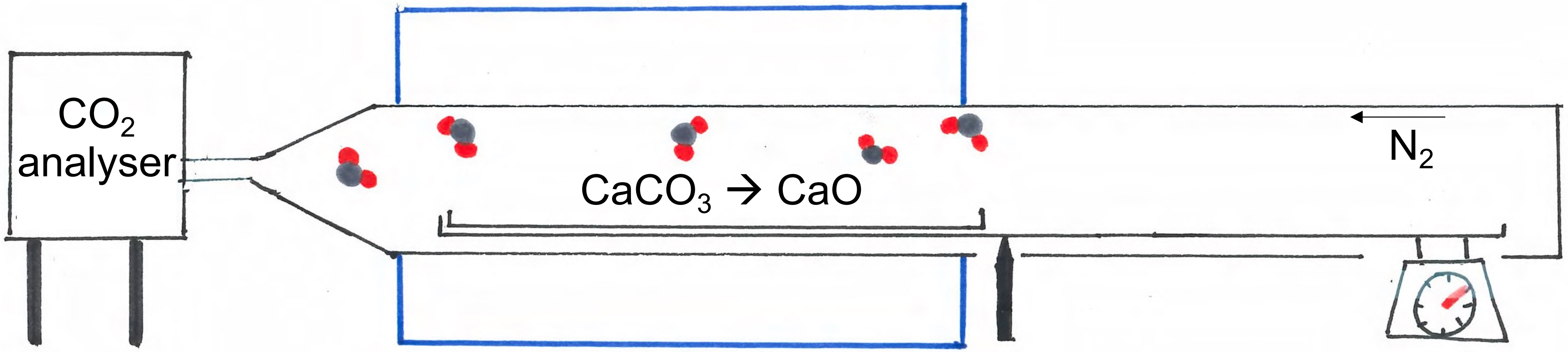
# Experiments

- Goal:
  - Demonstrate balance is working
  - Compare with TGA data
- $CaCO_3 \rightarrow CO_2 + CaO$
  Single reaction, releasing only $CO_2$







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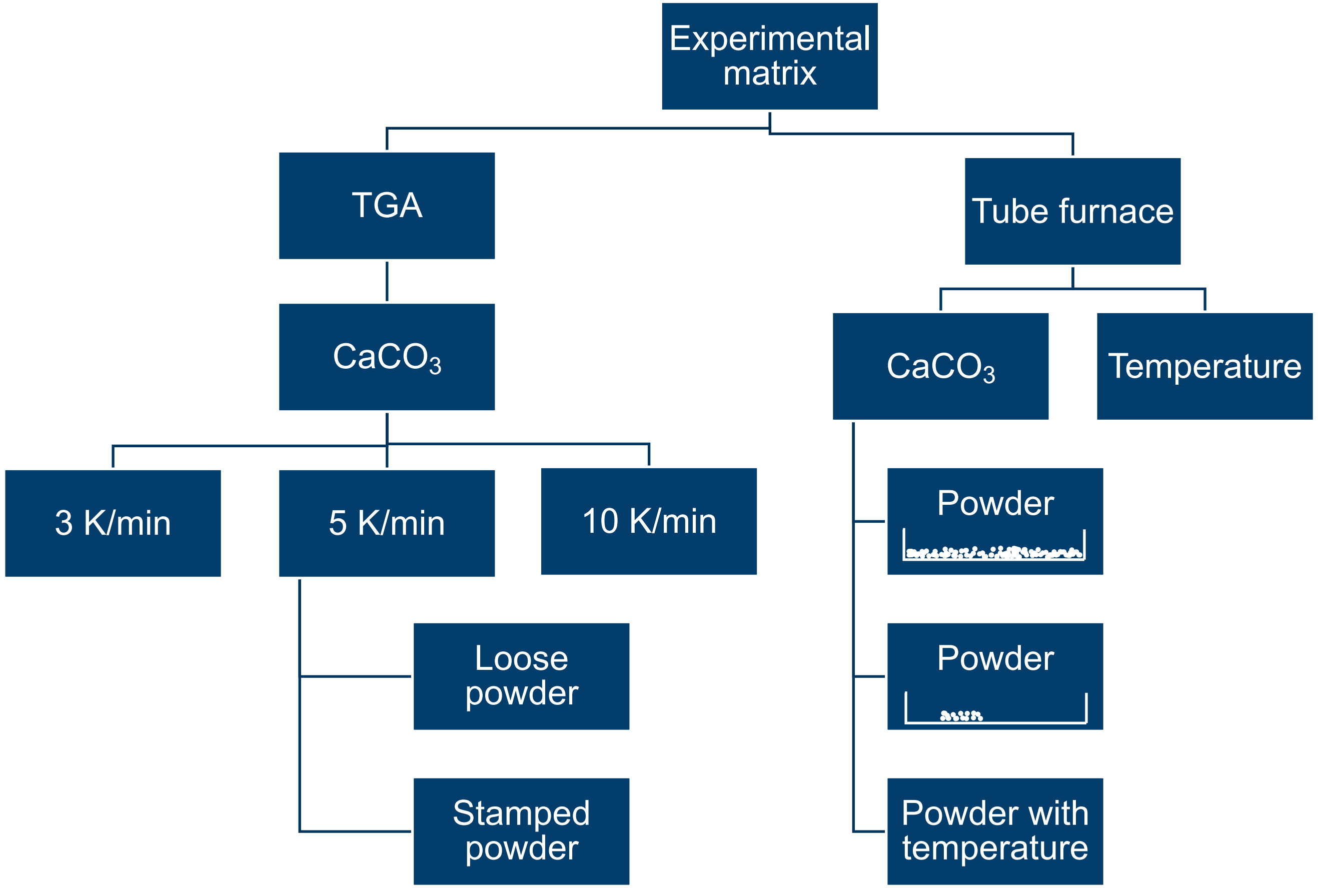
# Experiments



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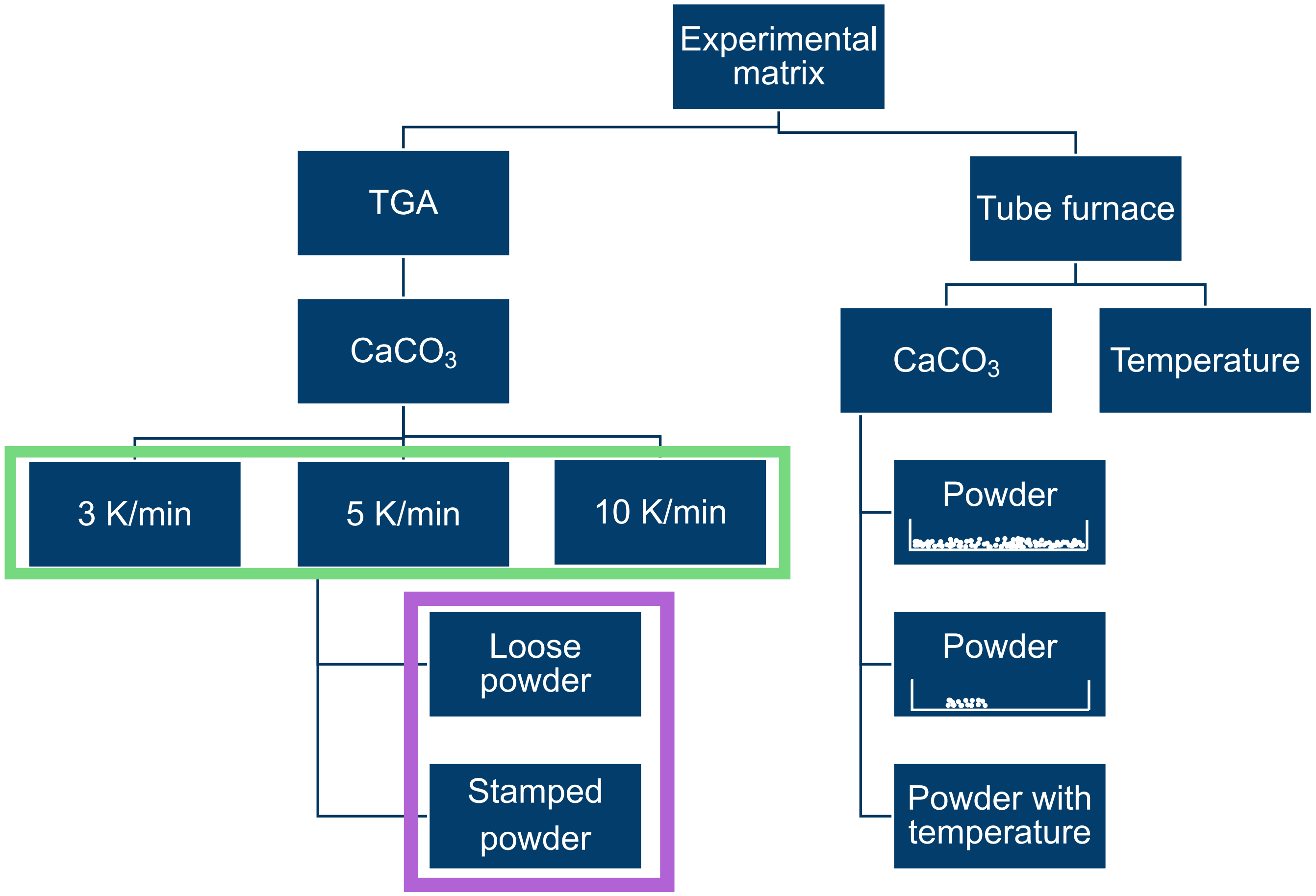
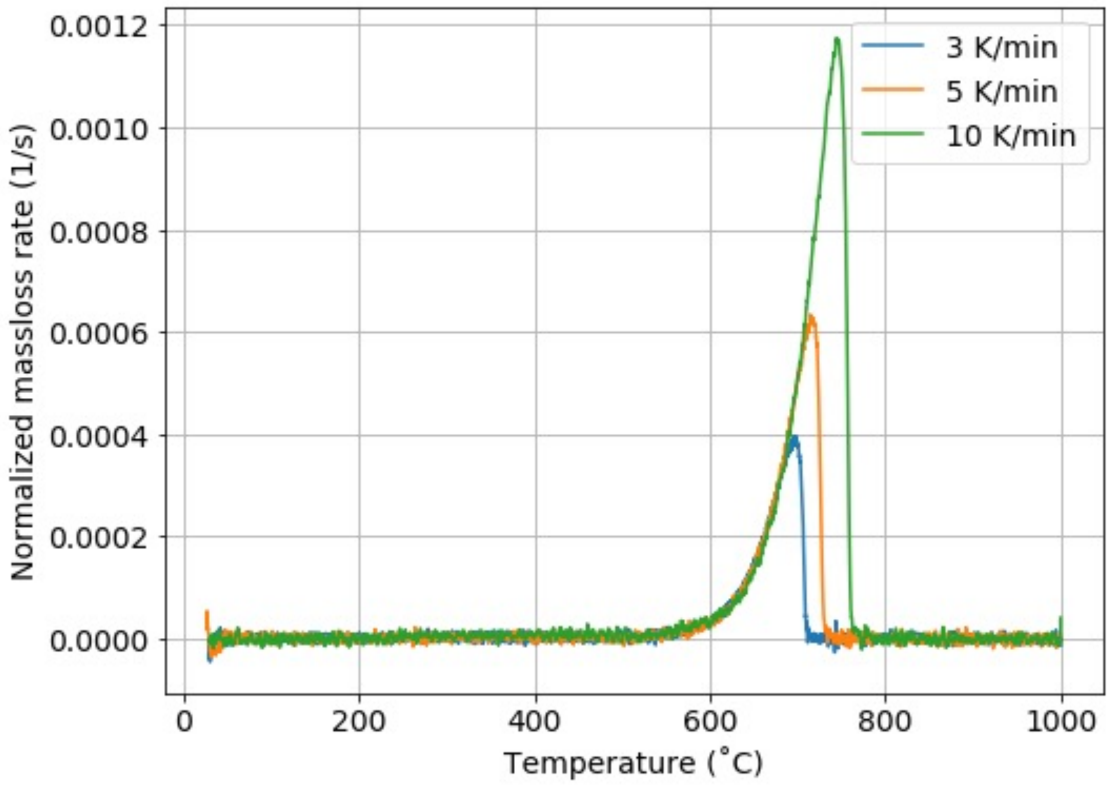
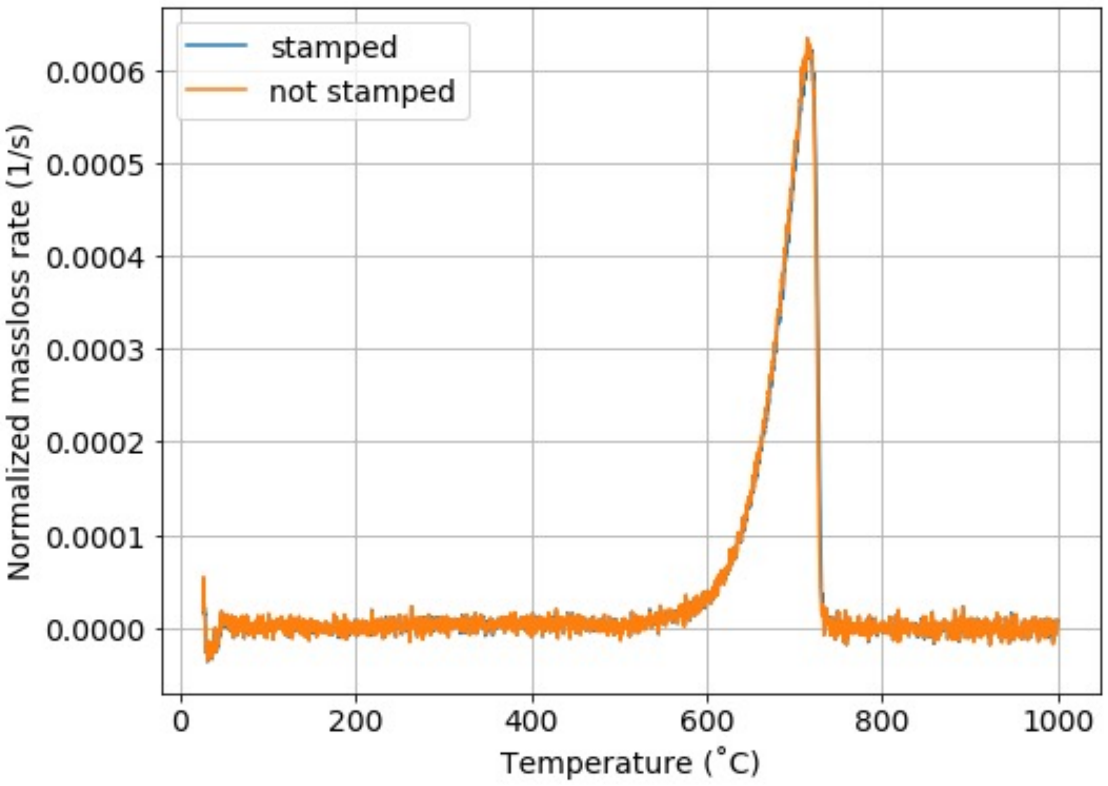
# Experiments



# Experiments

Averages of 3 repetitions



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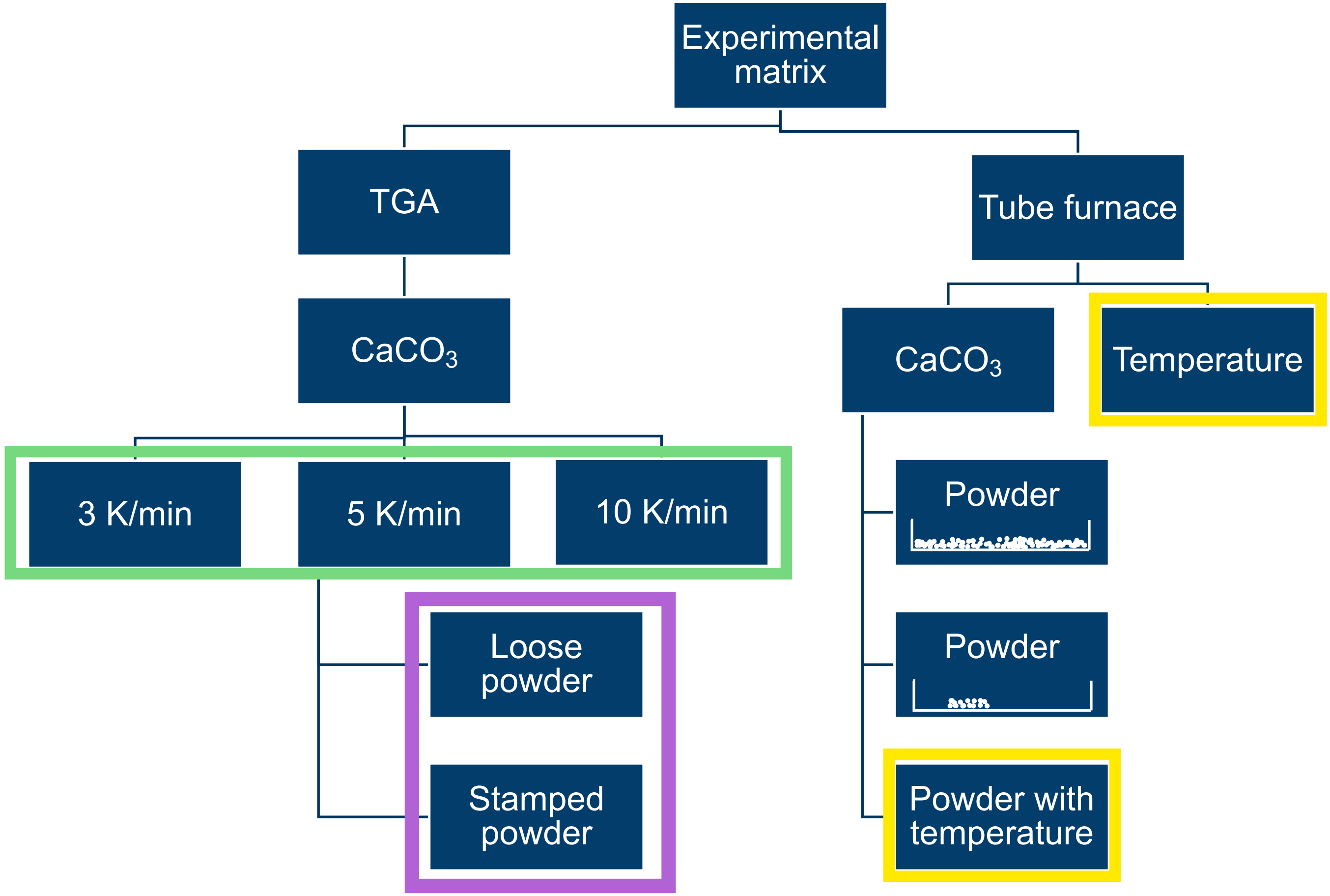
# Experiments



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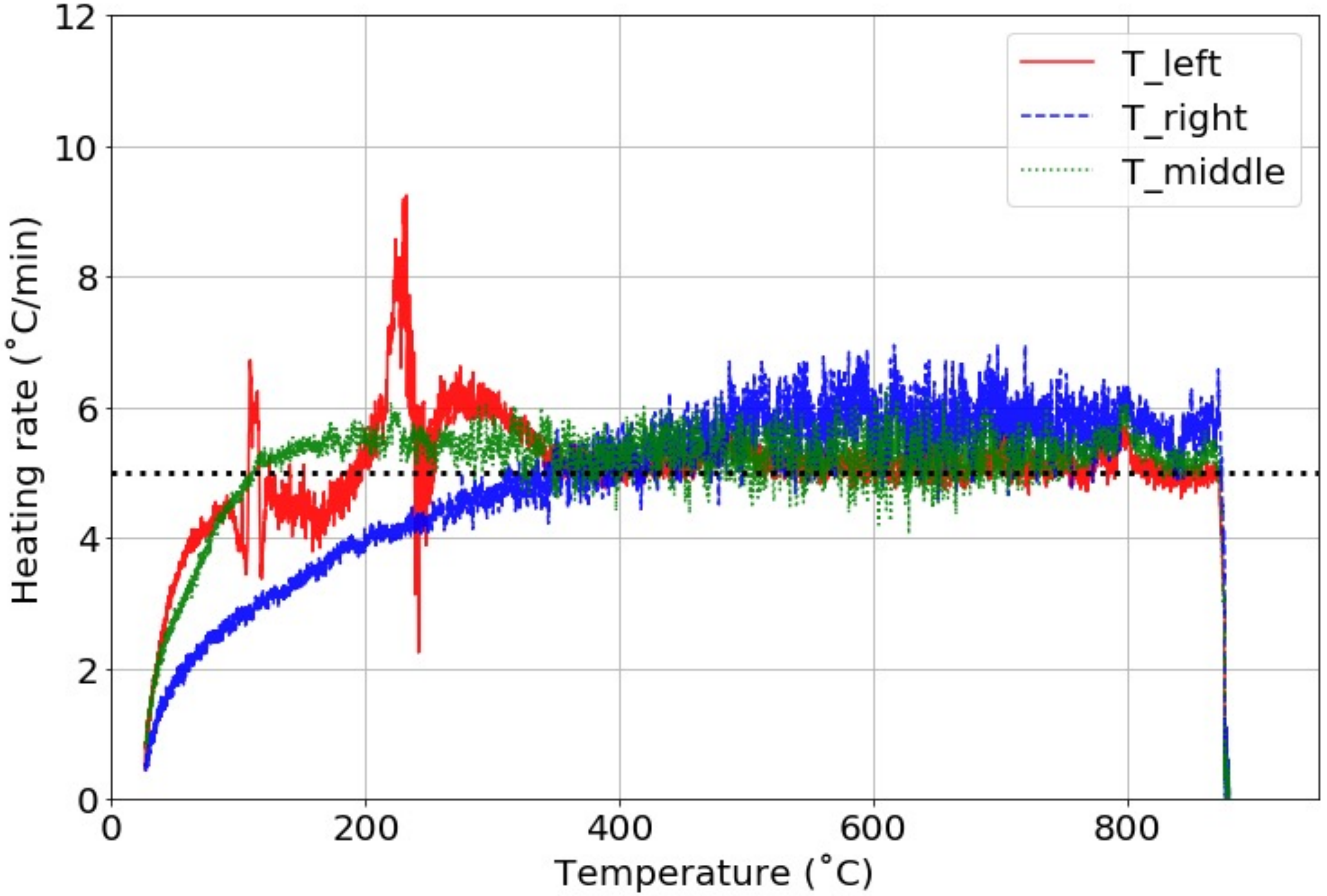


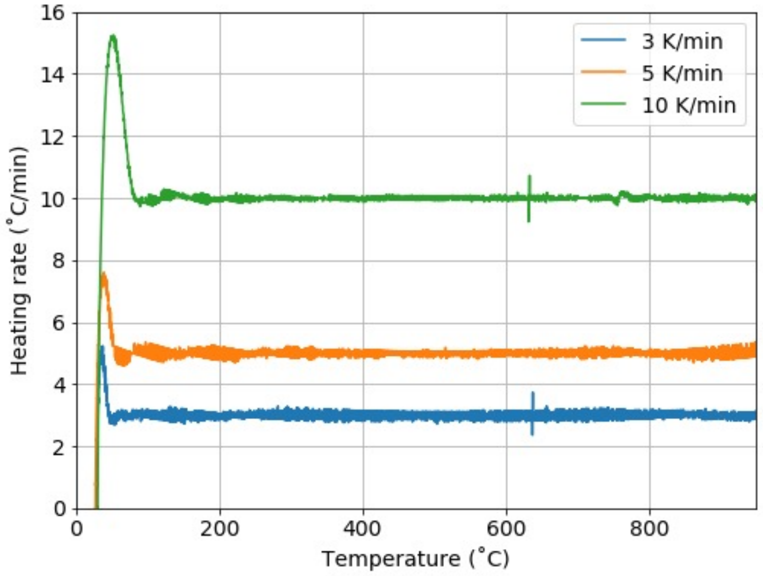


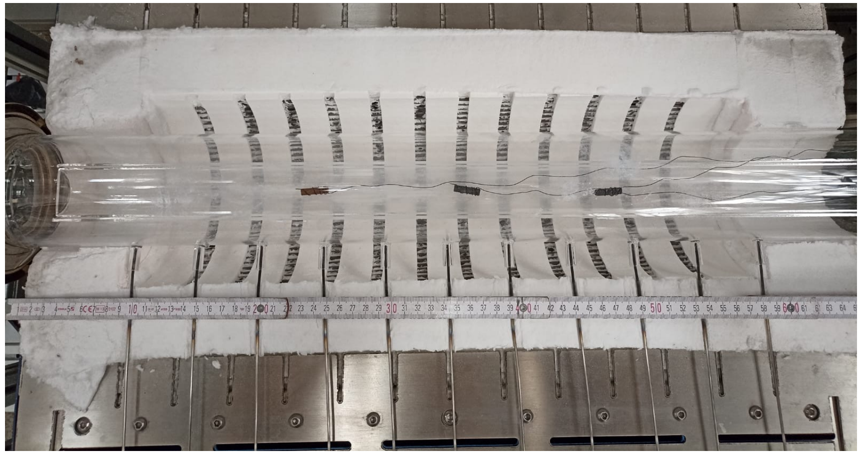



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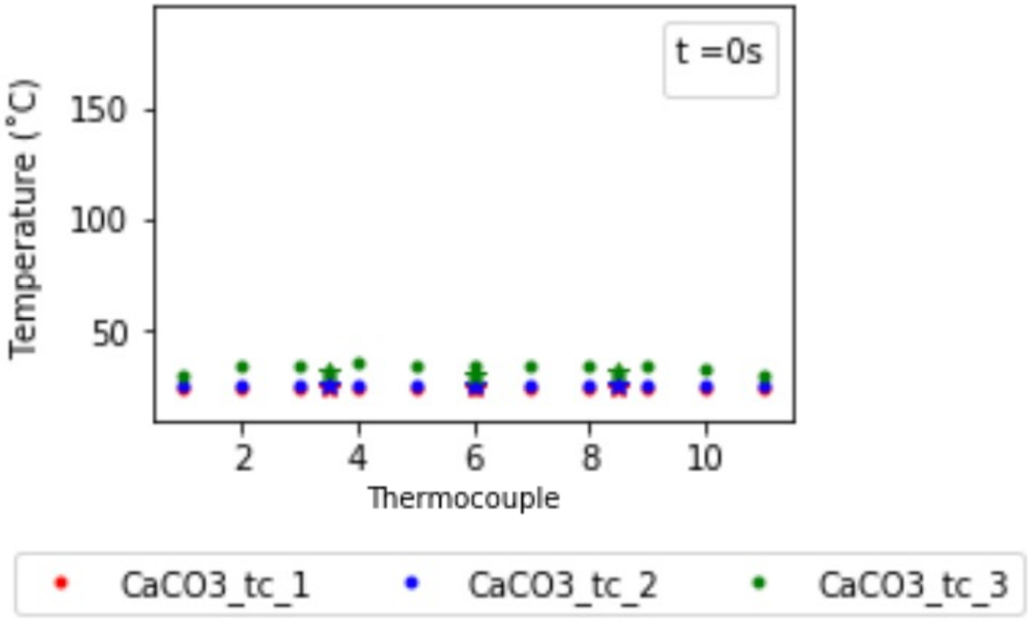
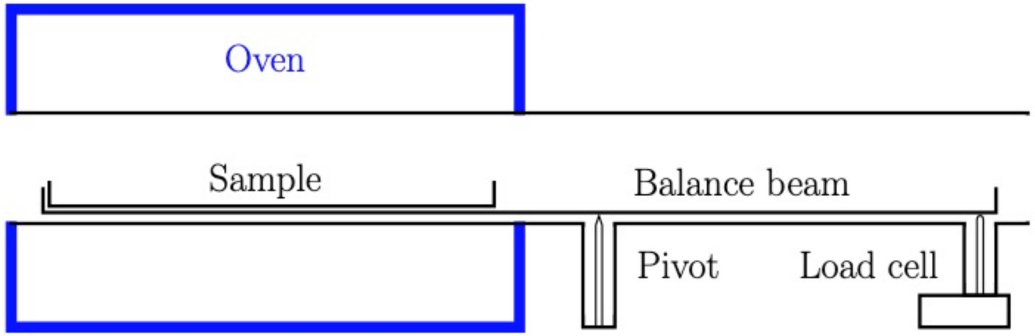
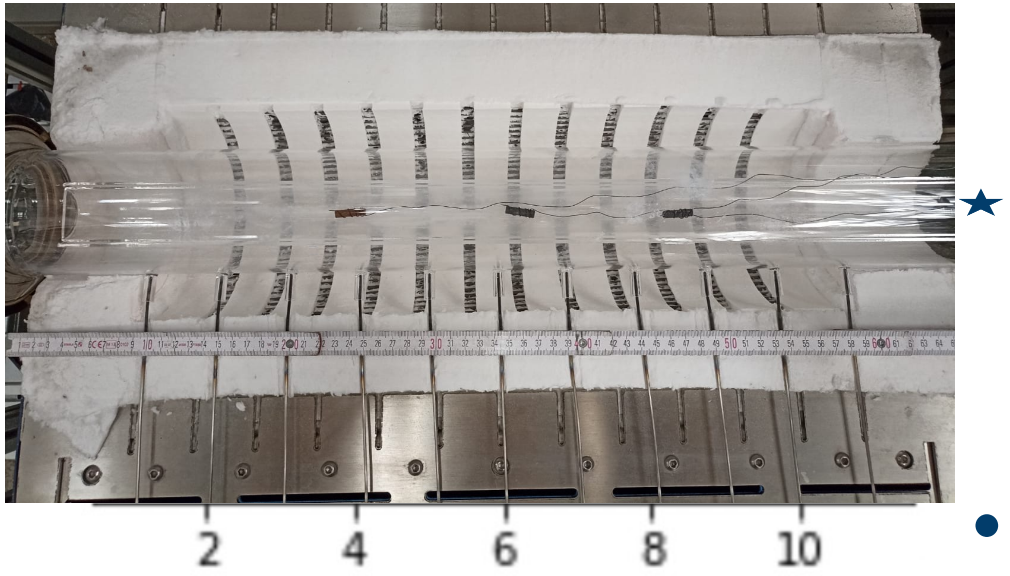
# Experiments



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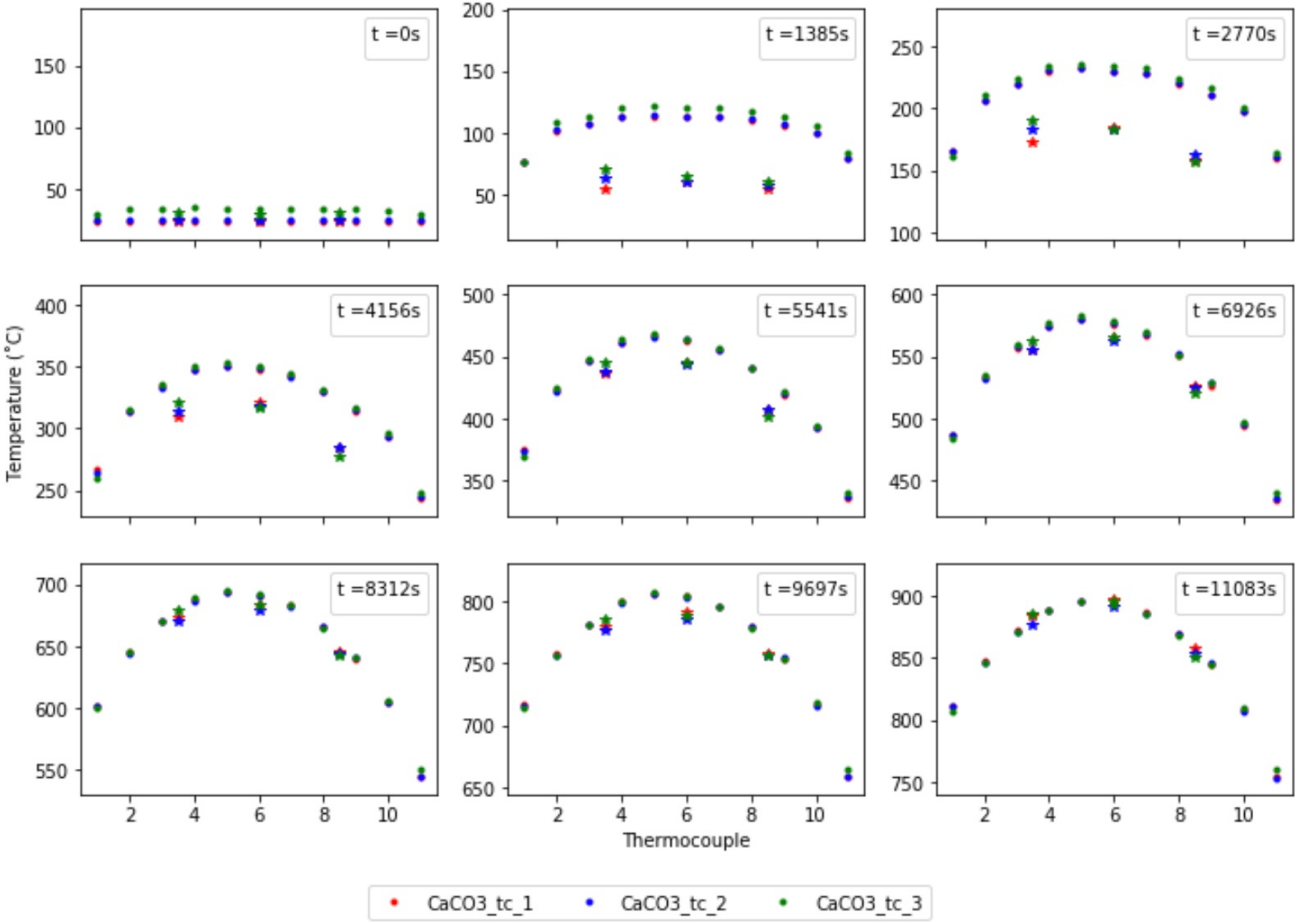
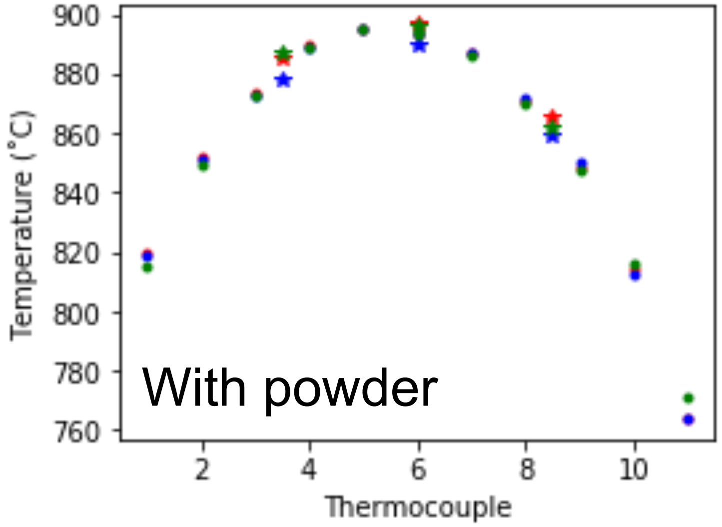
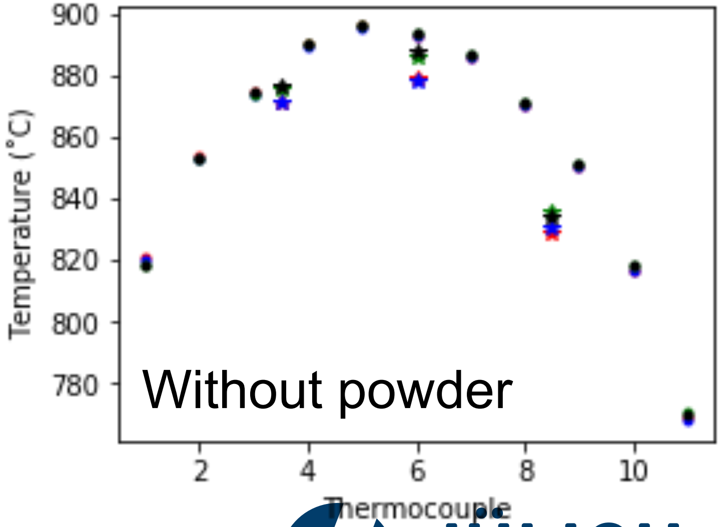
# Experiments

With powder




60 min at 900°C



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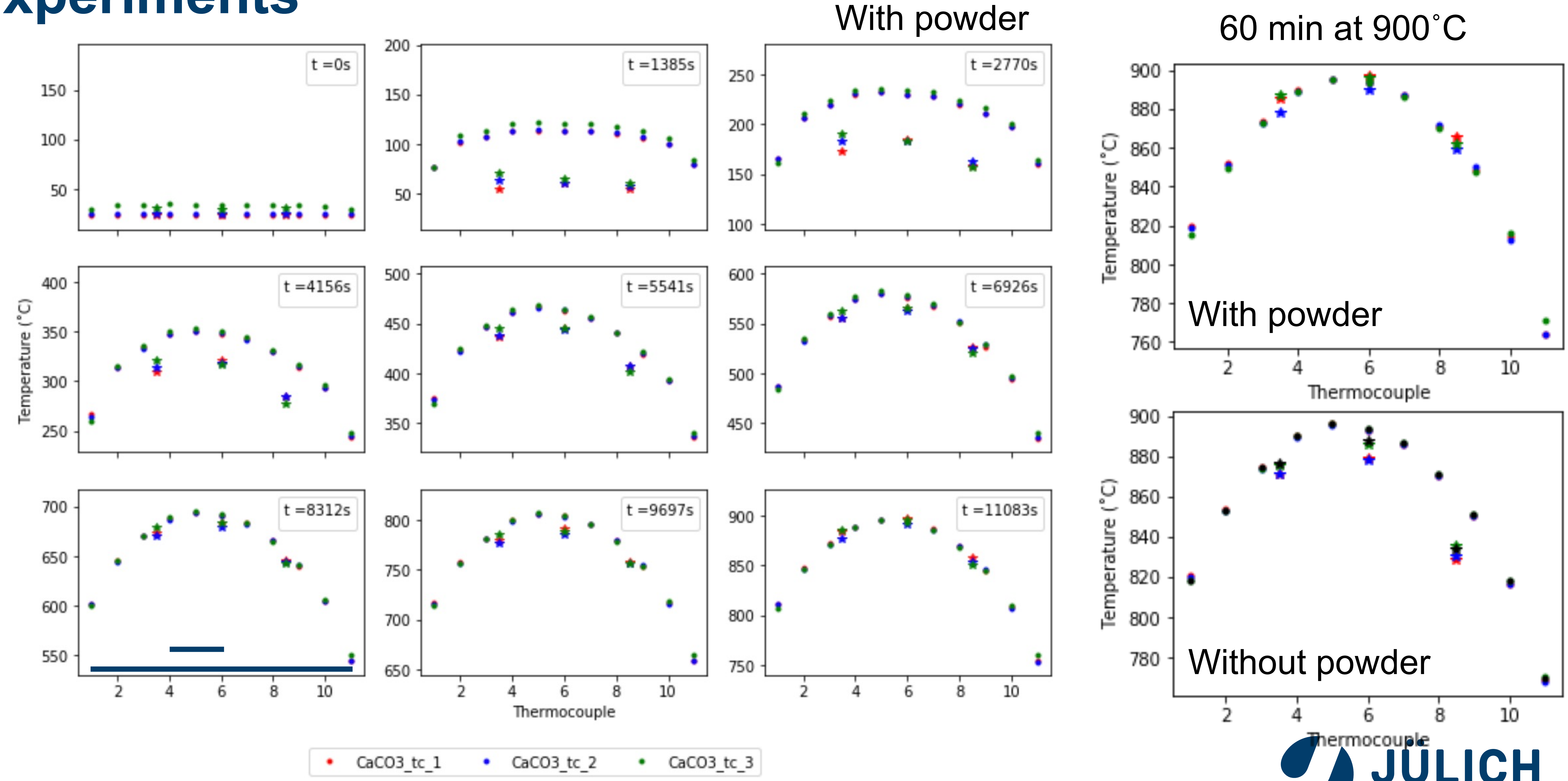
# Experiments



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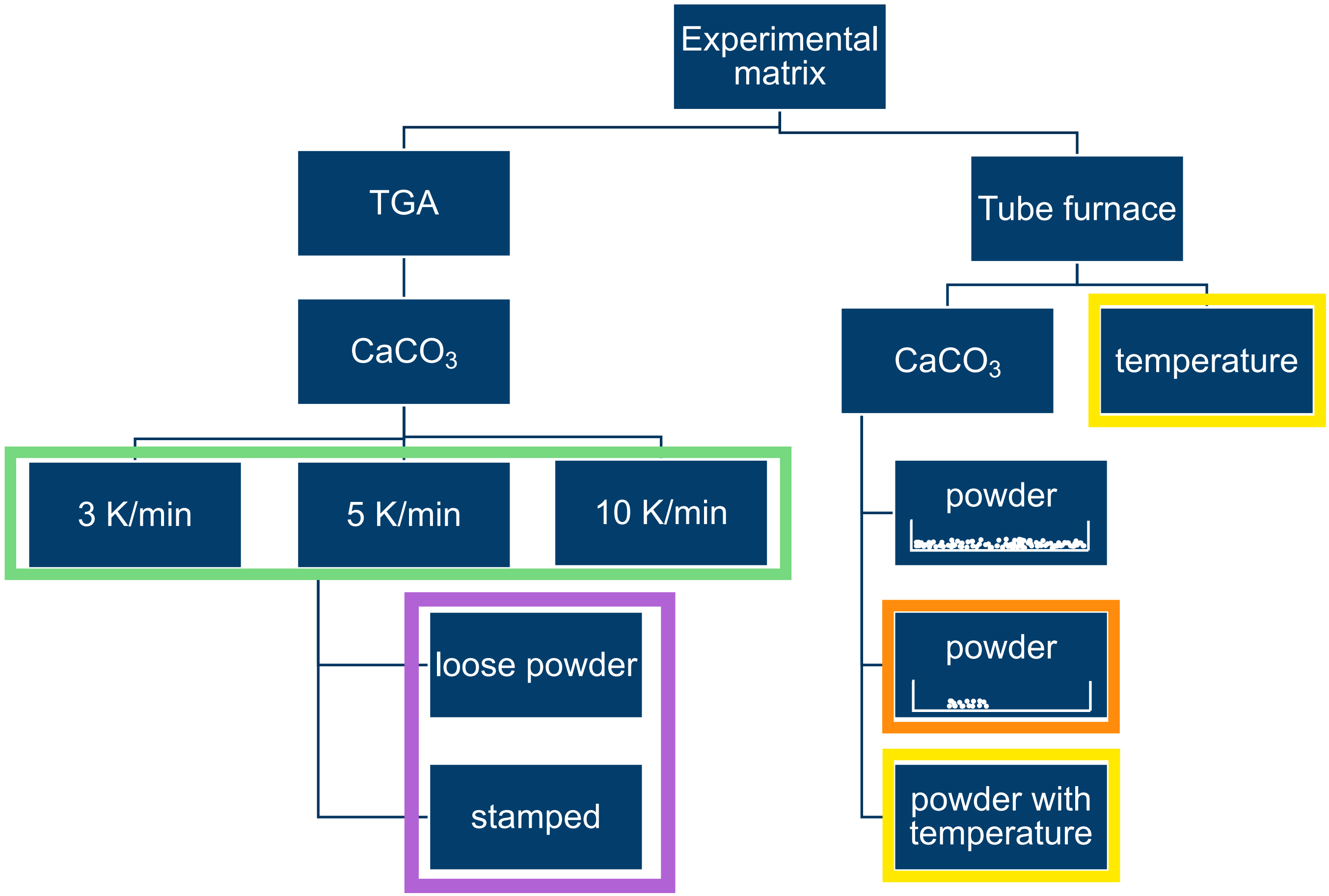
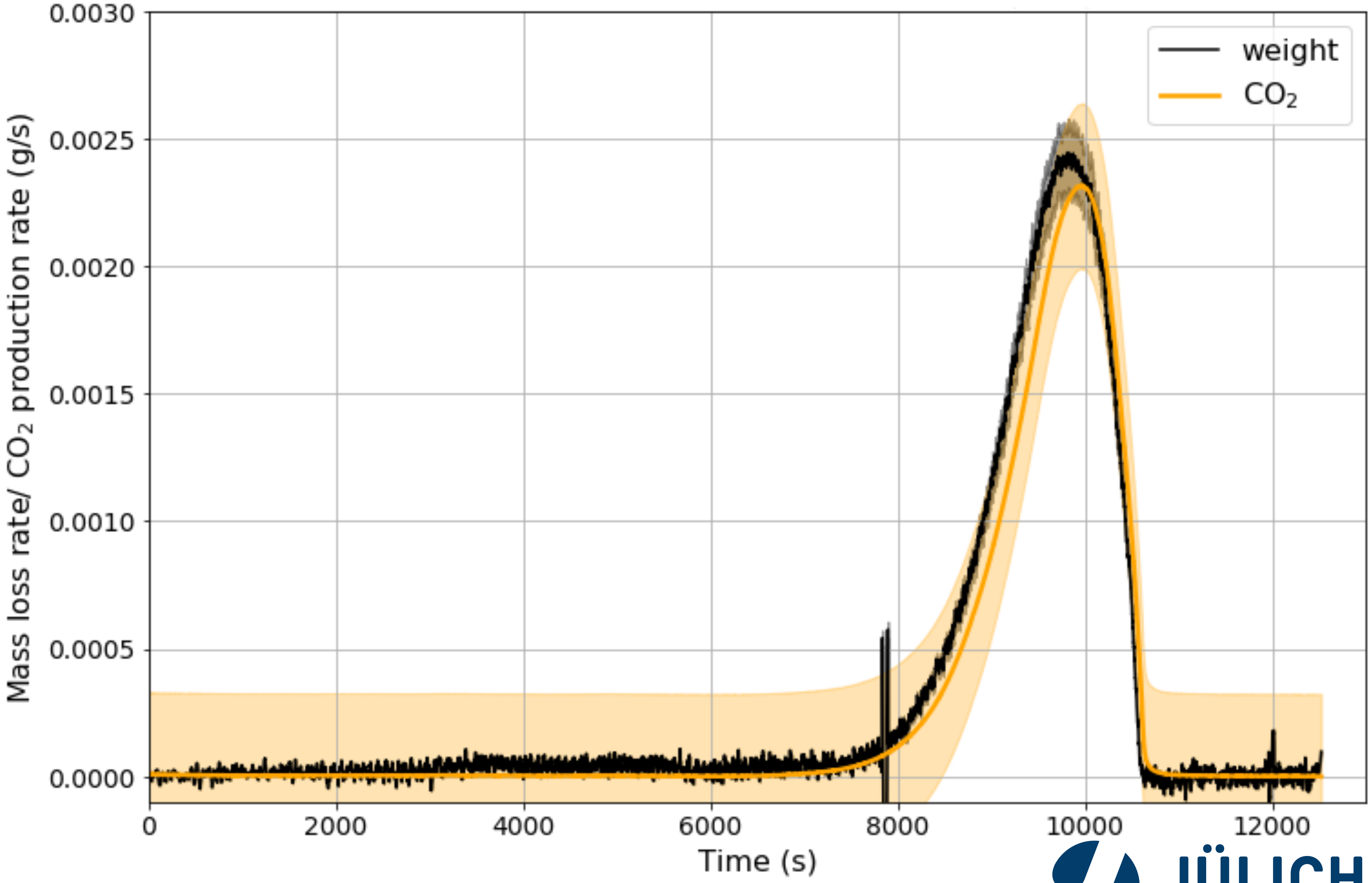
# Experiments

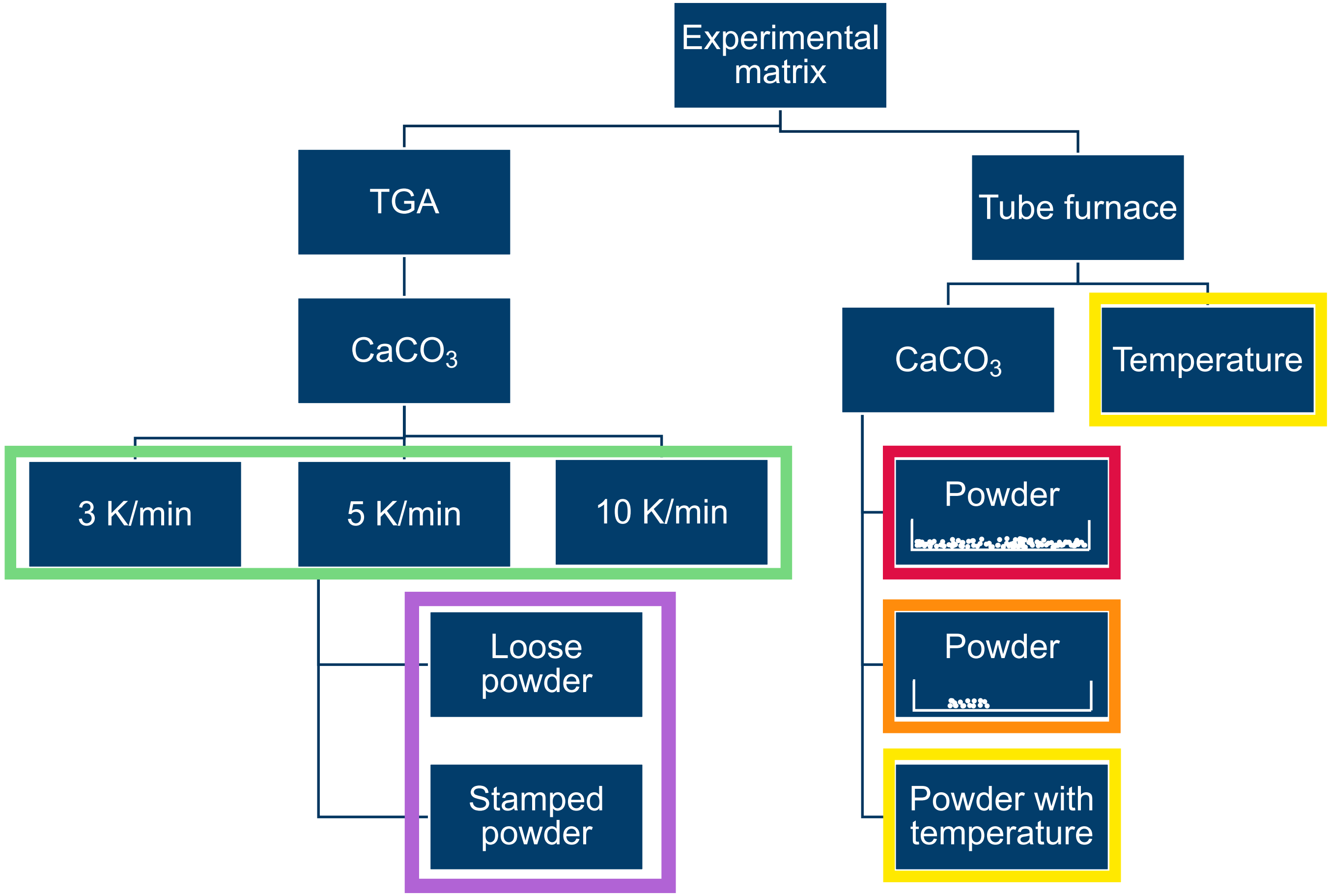


# Experiments

- Corrected with baseline
- Gas analyser uncertainty: given by manufacture
- Balance uncertainty: 0.1 g on start and end mass

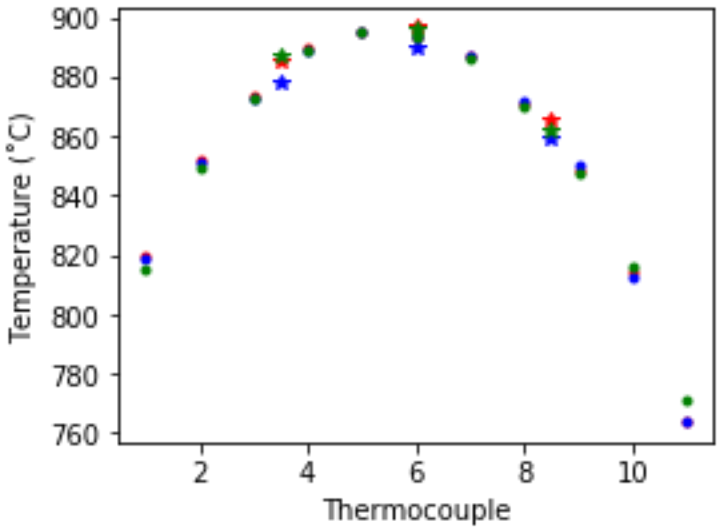


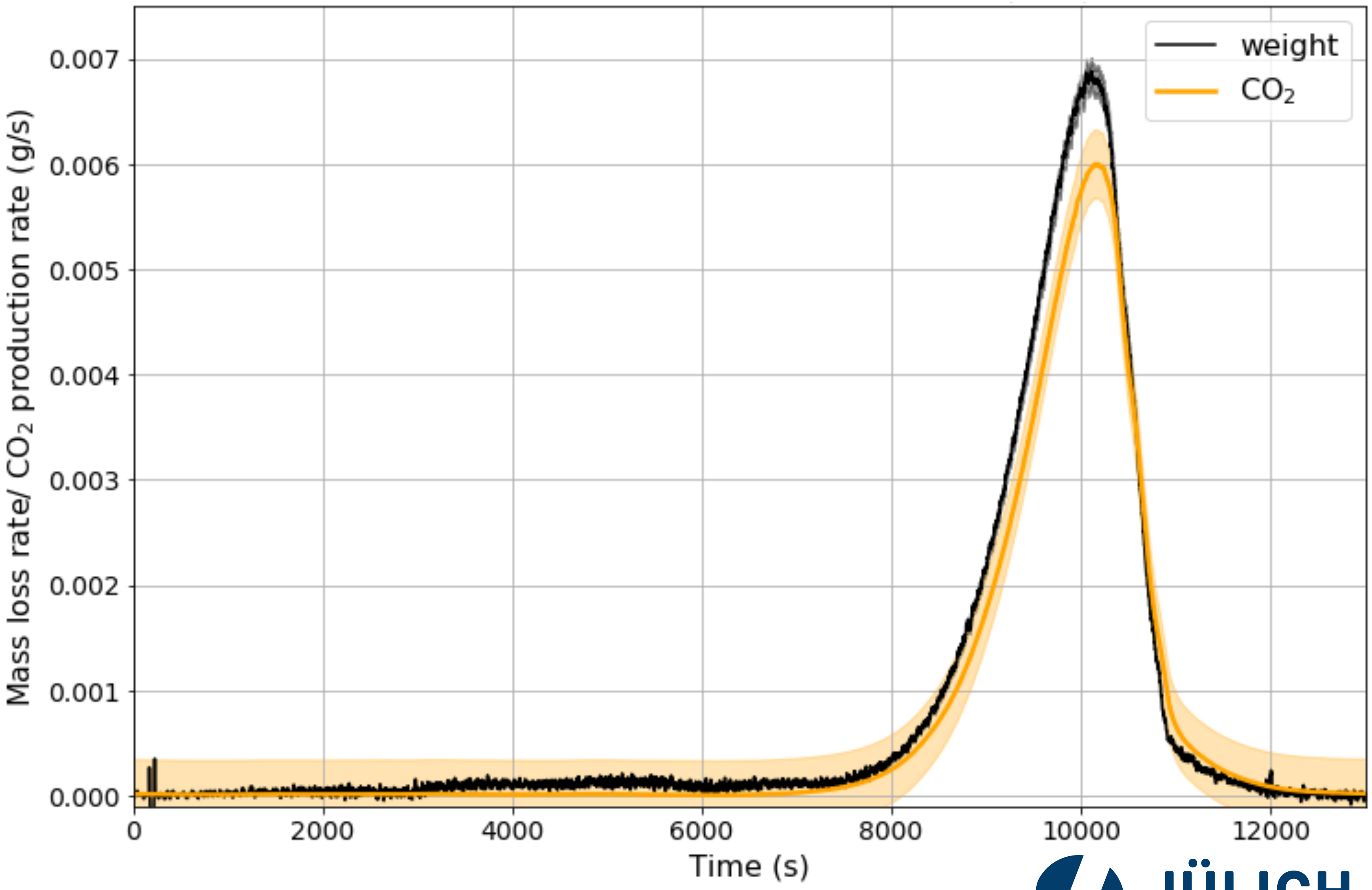
# Experiments



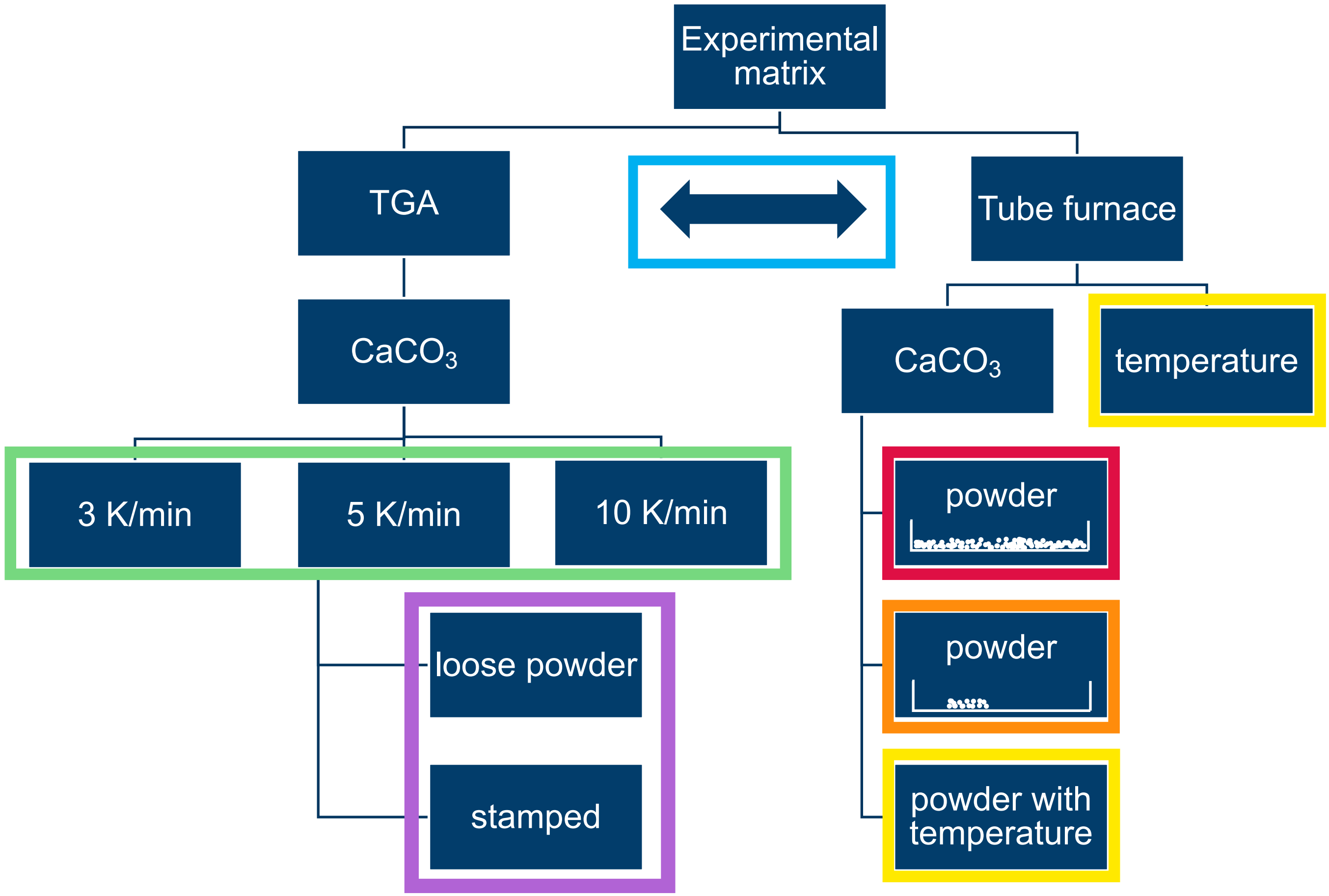
# Experiments

- Deviation due to unsymmetrical mass loss wrt centre of mass



# Experiments



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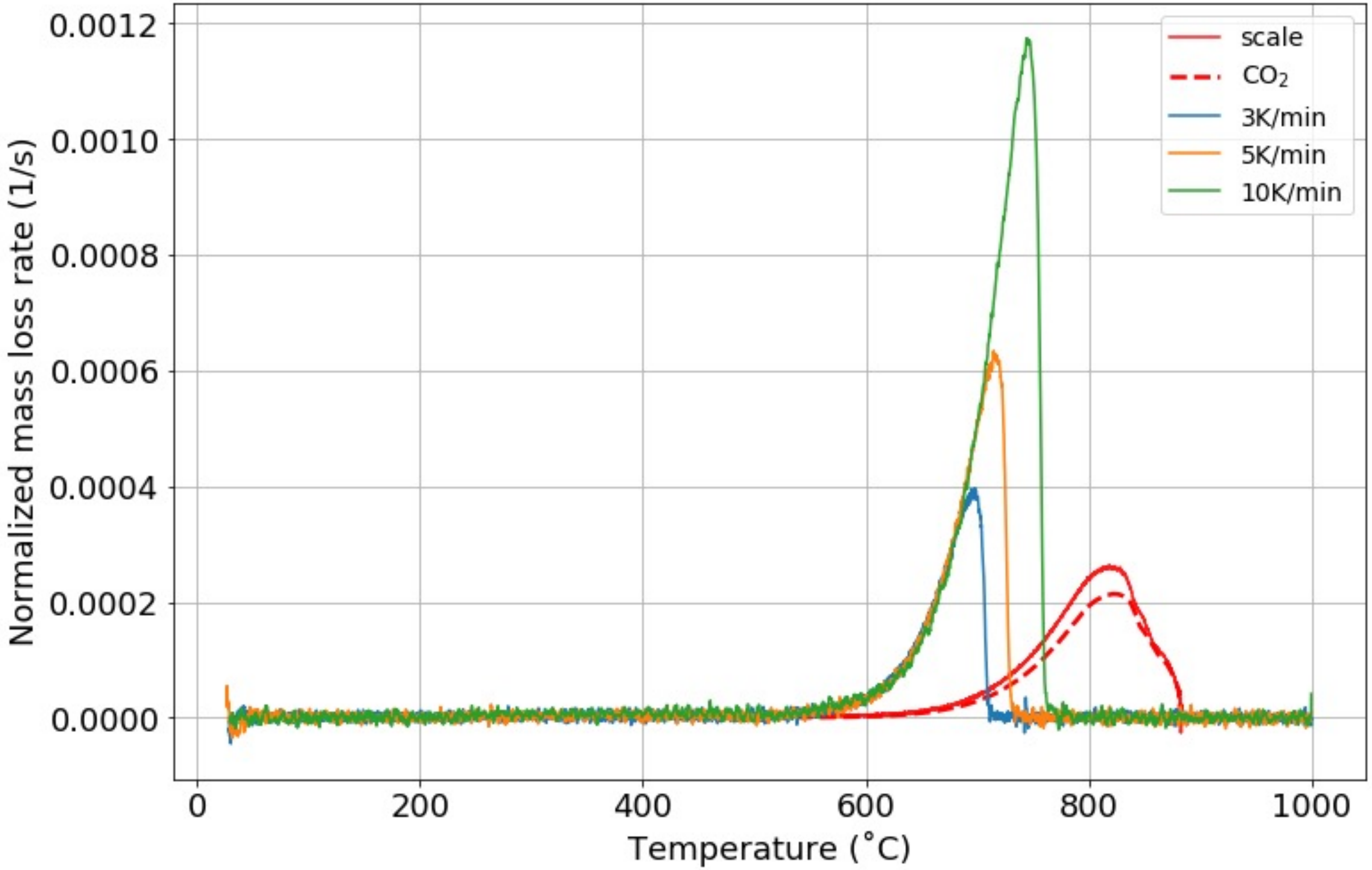
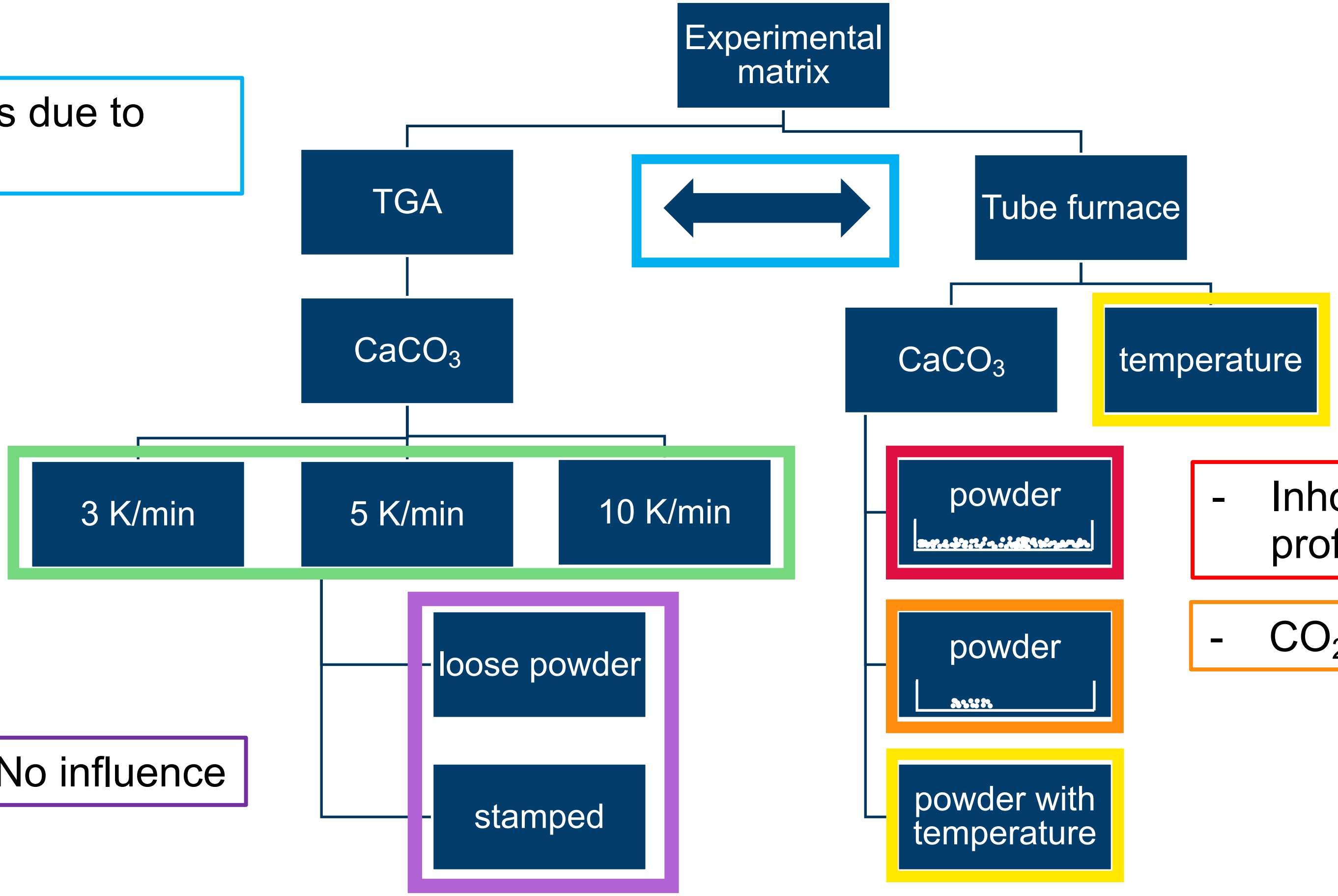
# Experiments



# Conclusion

Shift to higher temperatures due to thermal lag

For higher heating rate:
- Larger max. mass loss rate
- Max. mass loss rate as higher T




No influence

- 5 K/min heating rate achieved
- Temperature inhomogeneity 130˚C

- Inhomogeneity in temperature profile oven influences mass loss

- $CO_2$ and balance results agree

# References

[1] NUREG/CR-7010, Vol. 1 Cable Heat Release, Ignition, and Spread in Tray Installations During Fire July 2012 (CHRISTIFIRE) Phase 1: Horizontal Trays

[2] Hehnen T, Arnold L, La Mendola S. *Numerical Fire Spread Simulation Based on Material Pyrolysis—An Application to the CHRISTIFIRE Phase 1 Horizontal Cable Tray Tests*. Fire. 2020; 3(3):33. https://doi.org/10.3390/fire3030033

[3] Lannon CM, Stoliarov SI, Lord JM, Leventon IT*. A methodology for predicting and comparing the full-scale fire performance of similar materials based on small-scale testing.* Fire and Materials. 2018;42:710–724. https://doi.org/10.1002/fam.2524

[4] Petrella, R.V. *The assessment of full-scale fire hazards from cone calorimeter data. Journal of fire science.* 1994;12:14-43. https://doi.org/10.1177/073490419401200102

[5] T.R. Hull, K. Lebek, M. Pezzani, S. Messa. *Comparison of toxic product yields of burning cables in bench and large-scale experiments*. Fire Safety J, 43 (2) (2008), pp. 140-150, 10.1016/j.firesaf.2007.06.004

[6] Fiola, G.J. et al; *Comparison of Pyrolysis Properties of Extruded and Cast Poly (methyl methacrylate*). Fire safety journal. 2021;120: 103083, https://doi.org/10.1016/j.firesaf.2020.103083.

[7] *Image from:* http://www.china-acrylicmirror.com/what-are-the-reasons-why-pmma-sheets-are-widely-used-in-the-lighting-industry.html, accessed 10-oct-2022

[8] Huang X, Nakamura Y (2020) *A review of fundamental combustion phenomena in wire fires*. Fire Technol 1–32. https://doi.org/10.1007/s10694-019-00918-5

[9] *LINSEIS STA PT1600 Thermowaage Bedienungsanleitung*, Linseis-Messgeräte. Selb.



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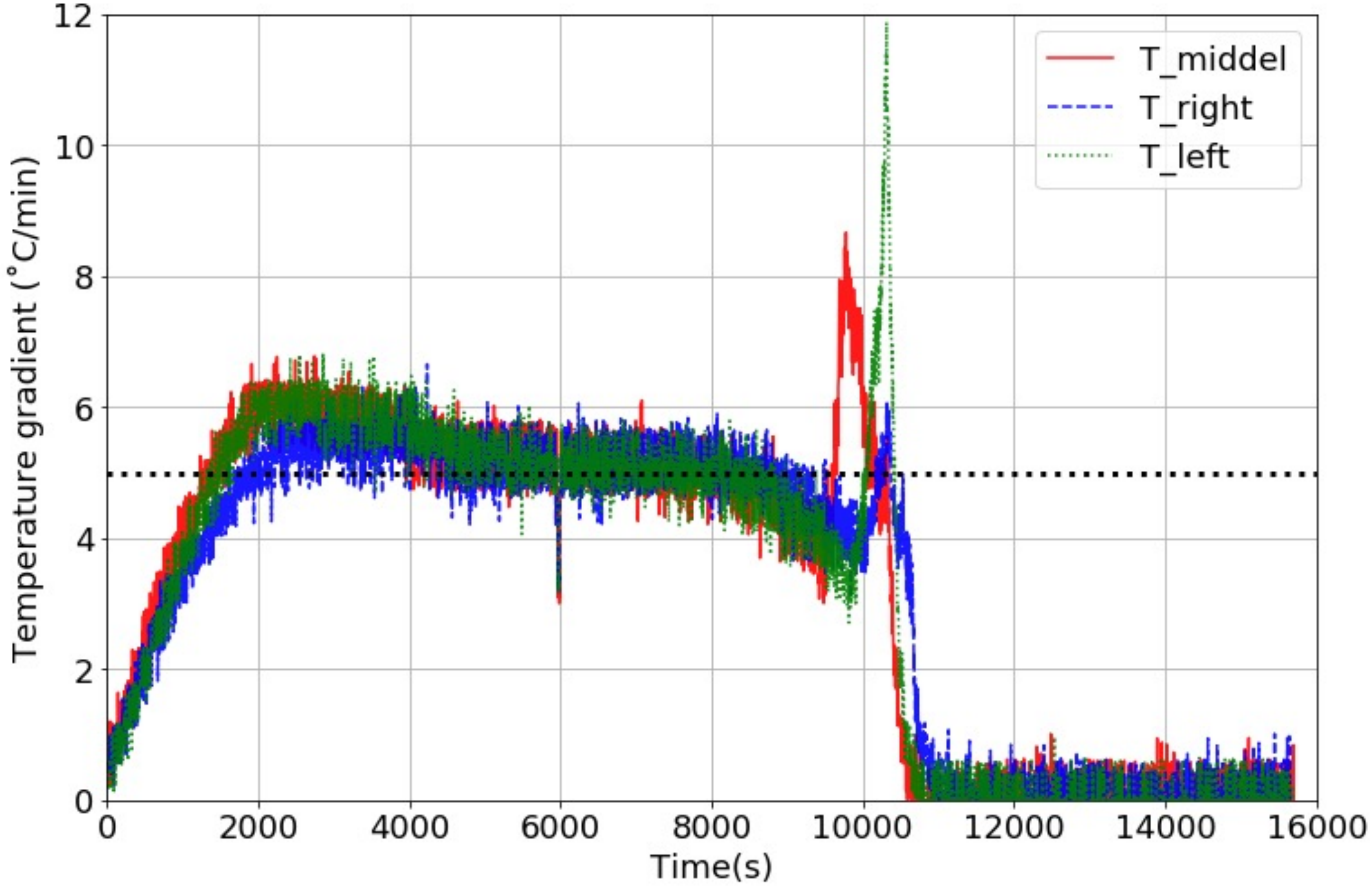
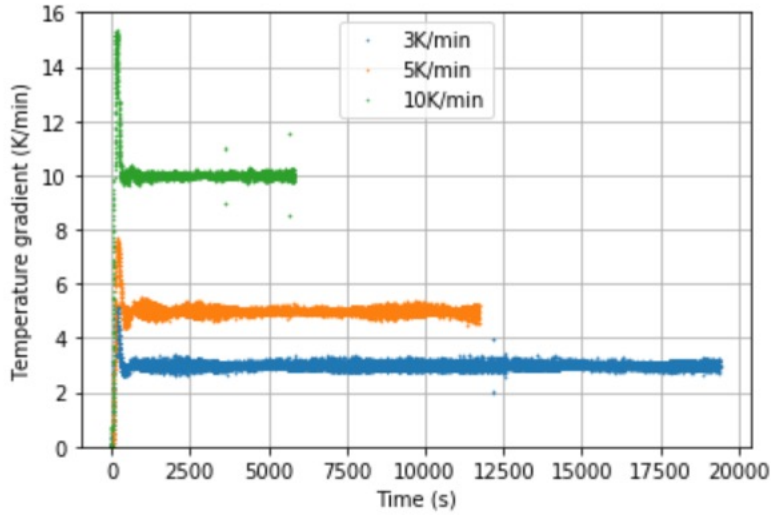
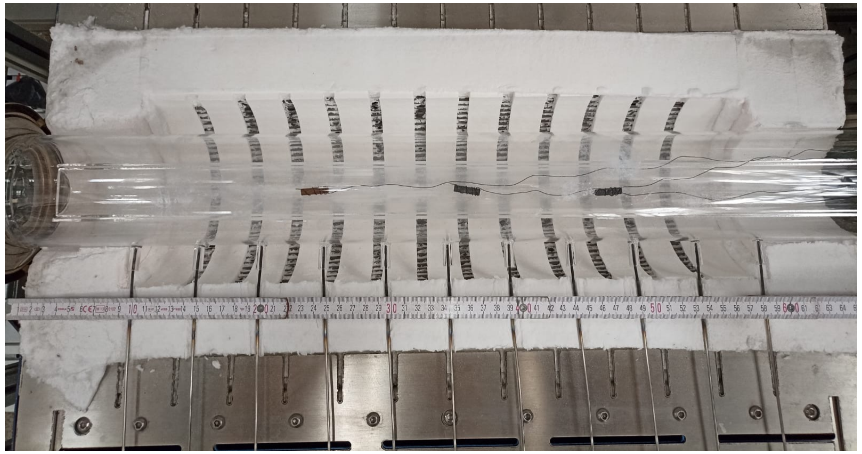
# Experiments





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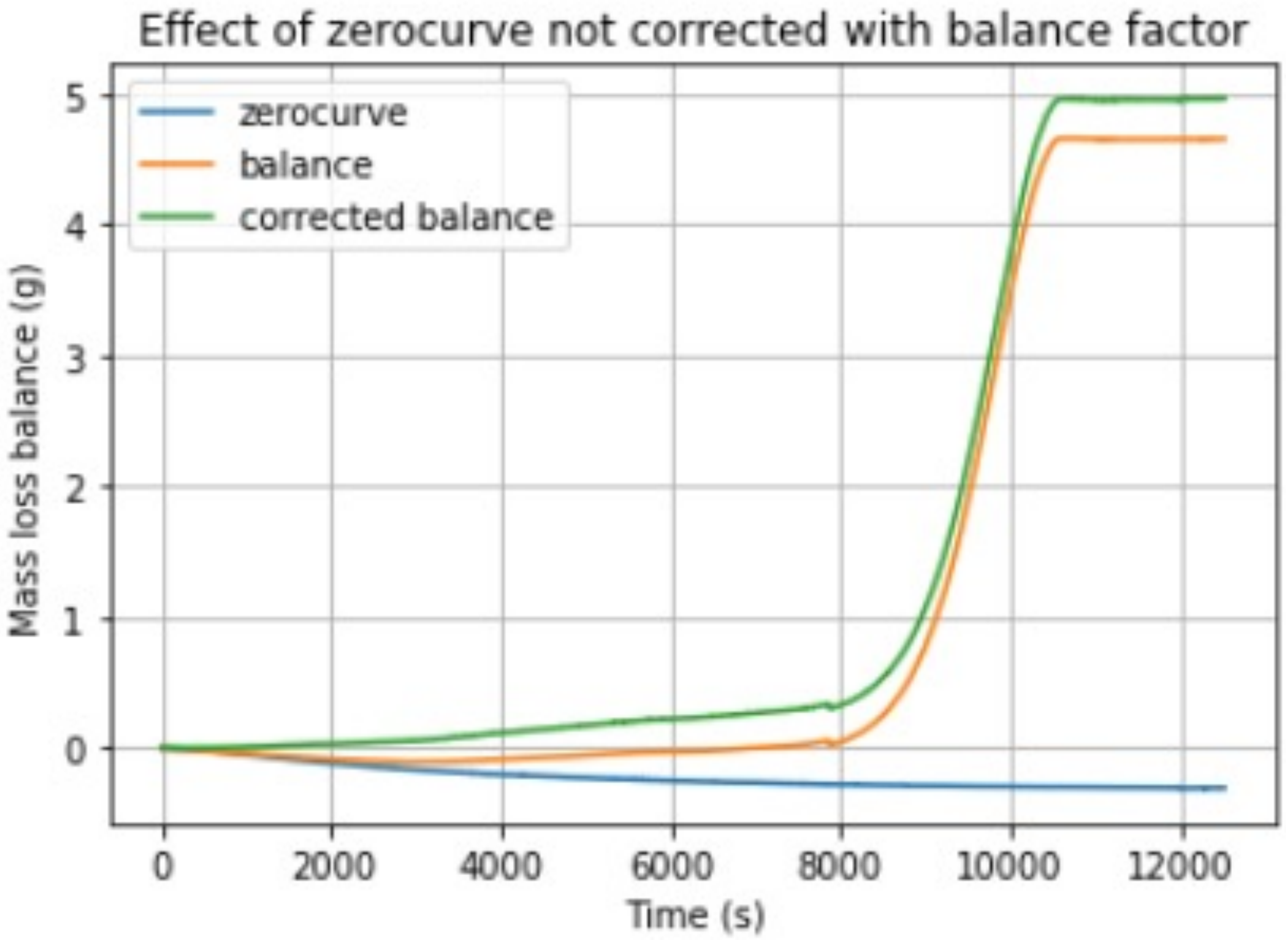
# DATA PROCESSING

## Zero curve correction



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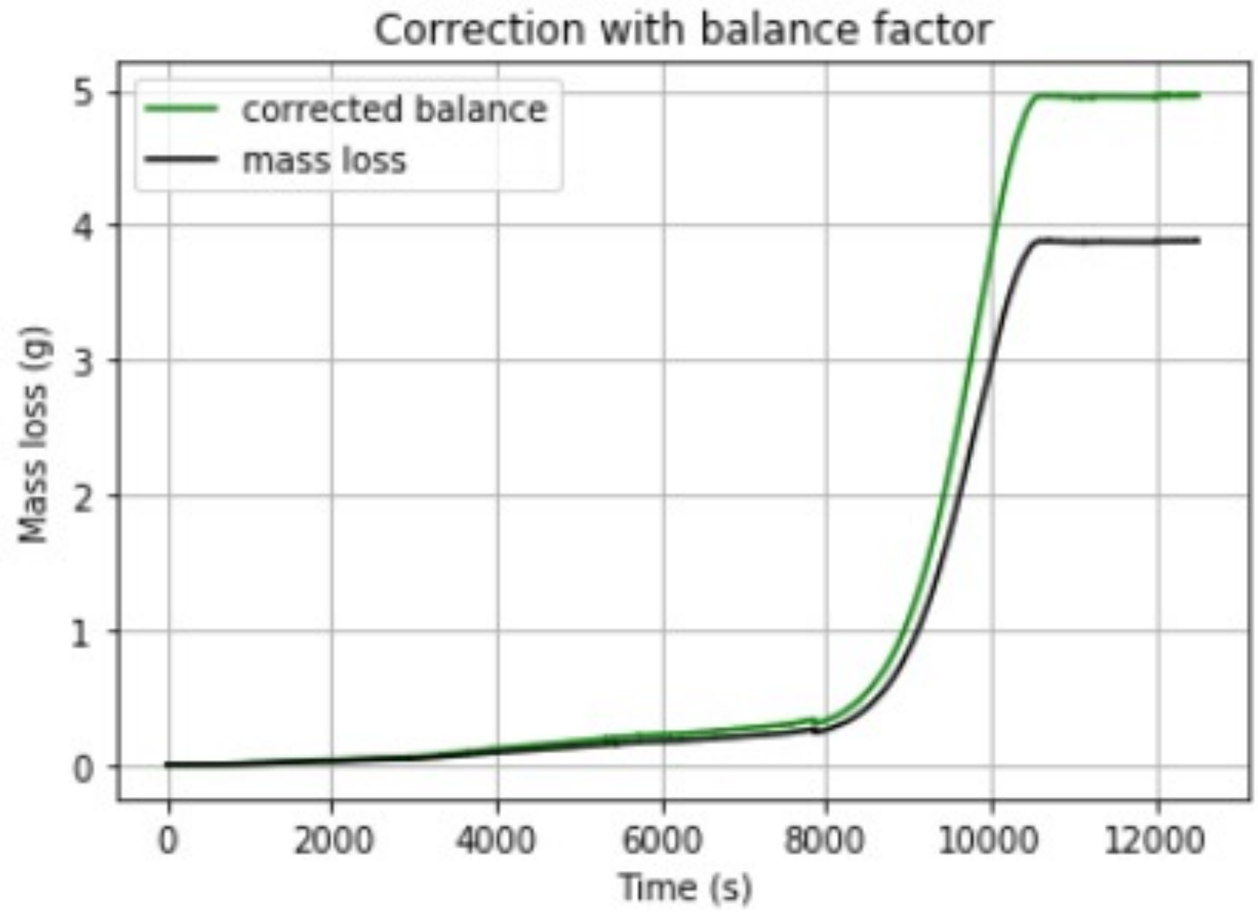
# Data processing

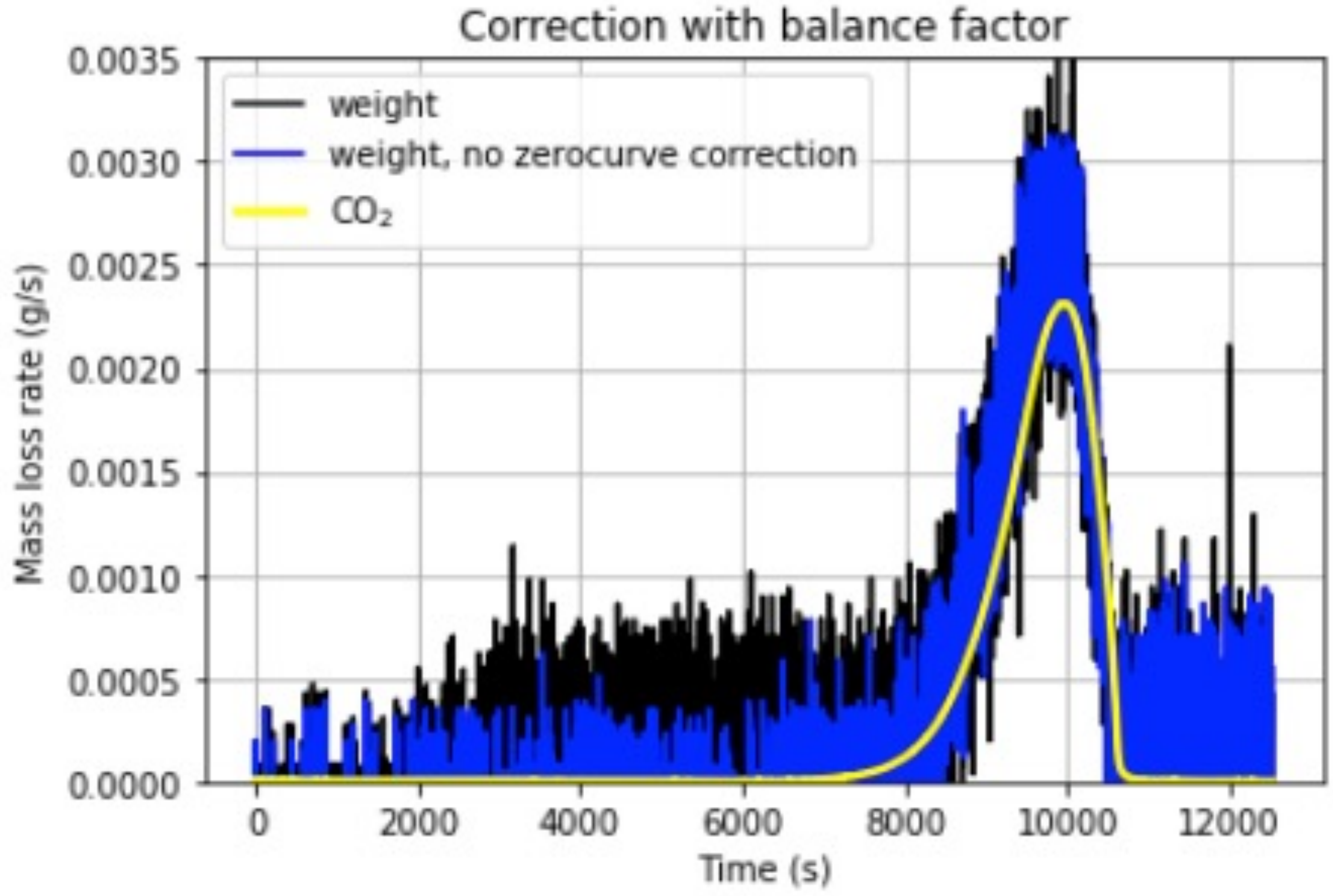



Balance factor:

$$factor = \frac{m_{start} - m_{end}}{m_{cell,start} - m_{cell,end}}$$

= 0.848

# Data processing




$\rho_{norm,N_2} = 1,234 \ kg/m^3$

$M_{N_2} = 28,0134 \cdot 10^{-3} \ kg/mol$

$M_{CO_2} = 44,01 \ g/mol$

$m_{flow,N_2} = \rho_{norm,N_2} \cdot 10 \cdot 10^{-3}$

$[kg/min]$

$\dot{n}_{N_2} = \frac{m_{flow,N_2}}{M_{N_2}} \quad [mol/min]$

$\dot{n}_{CO_2} = Vol\%CO_2 \cdot \dot{n}_{N_2} \quad [mol/min]$

$\dot{m}_{CO_2} = \frac{\dot{n}_{CO_2} \cdot M_{CO_2}}{60} \quad [g/s]$



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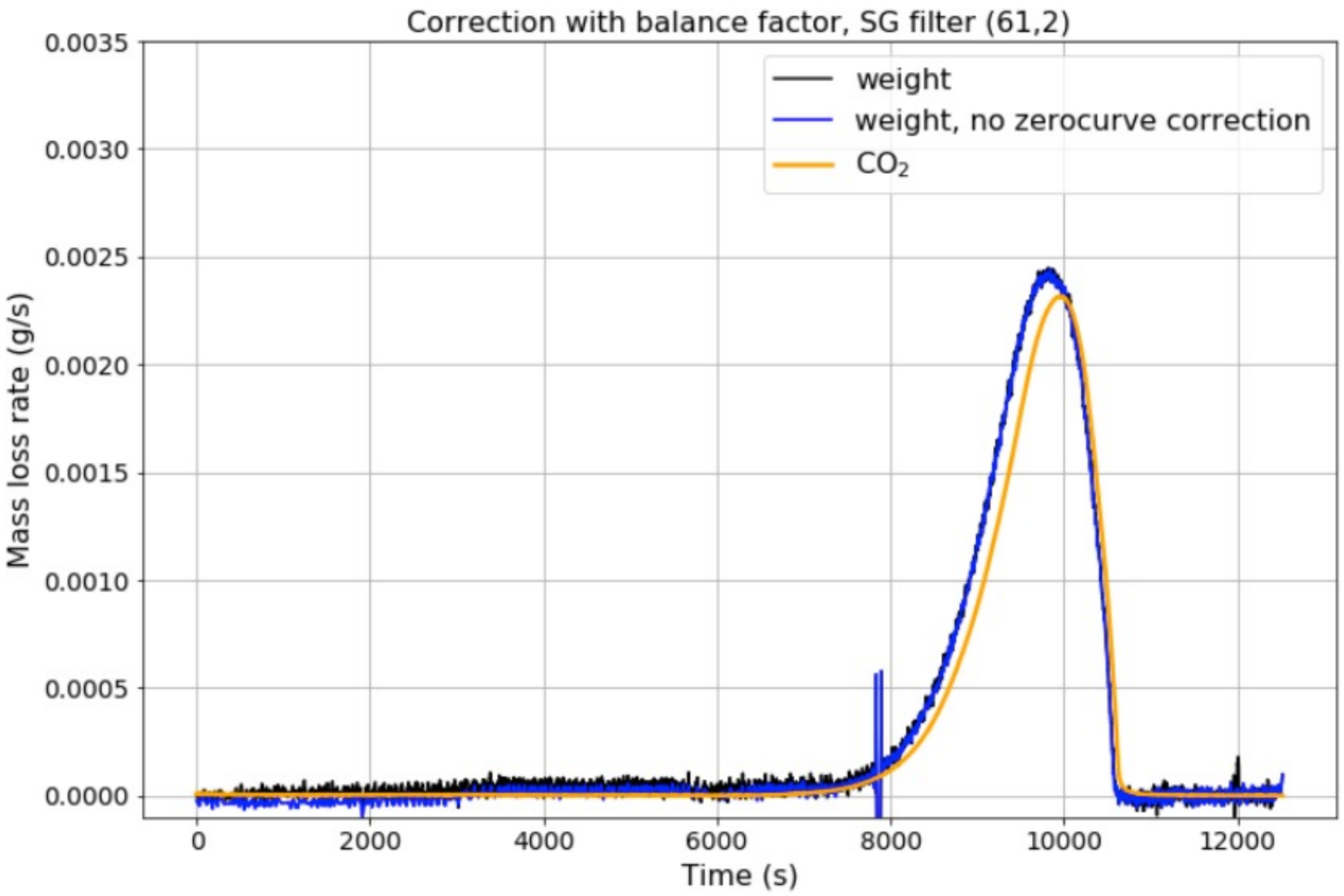
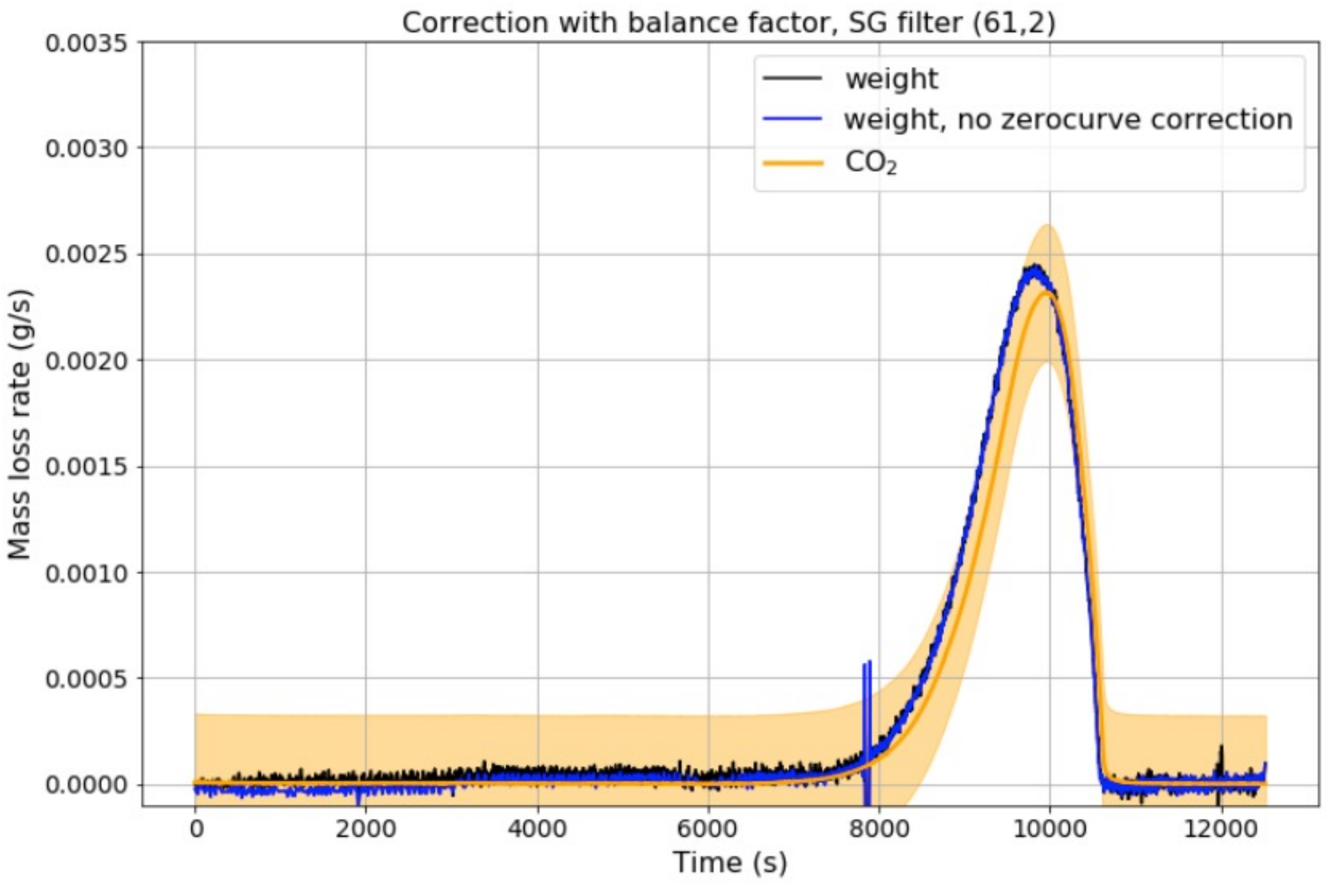
# Data processing




Mass loss: 3.64 g
Mass loss starting from 7000s: 3.44 g
Mass loss over CO2: 3.17 g



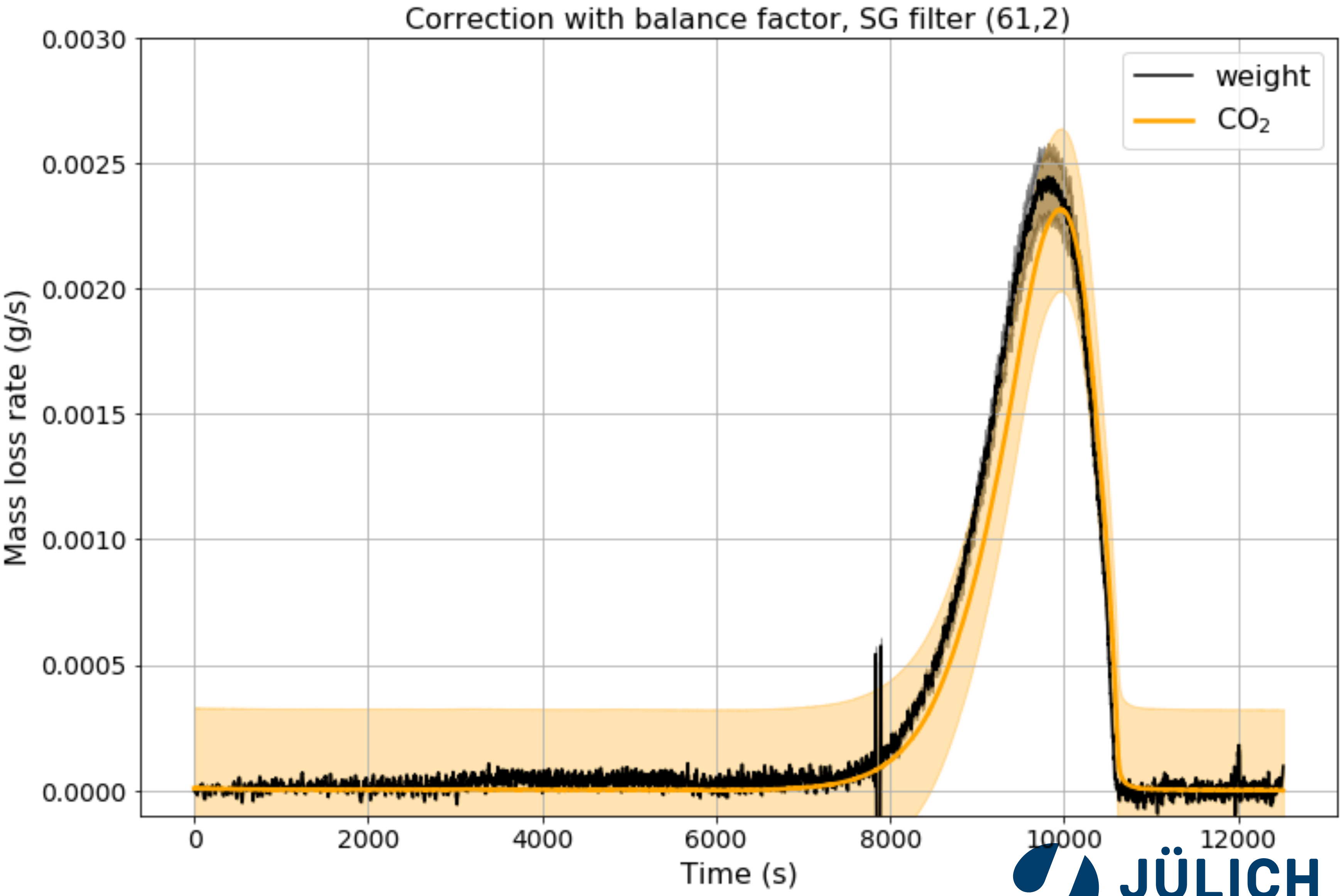

- Uncertainty on CO2:
  - 1% of maximum callibration
  - 0.1 Vol% of Co2

# Start mass - end mass

Start mass: 8.61 g
End mass: 4.97 g

Limits:
Up:
- start +0.1g
- end -0.1g

Lower:
- start -0.1g
- end +0.1g



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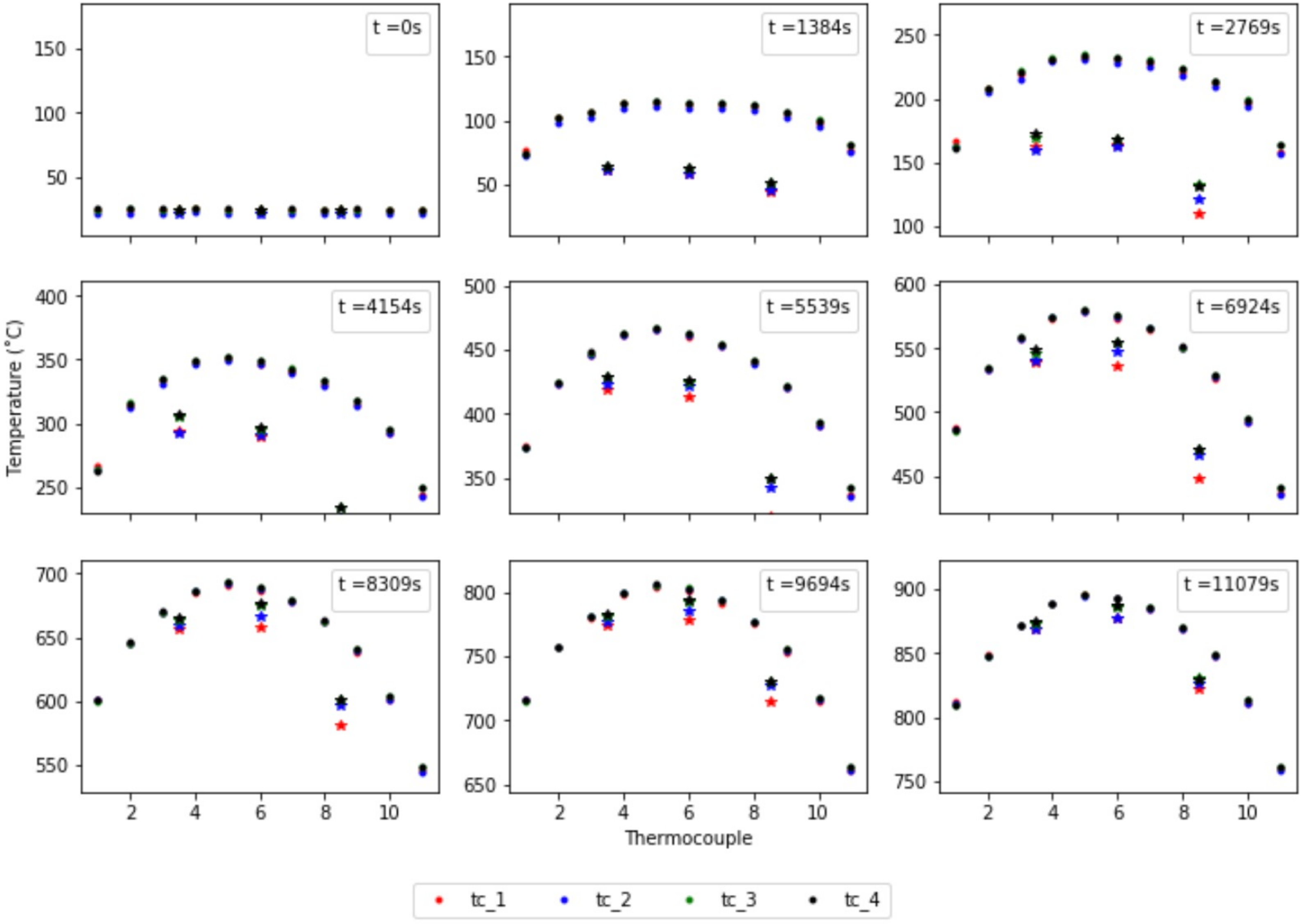
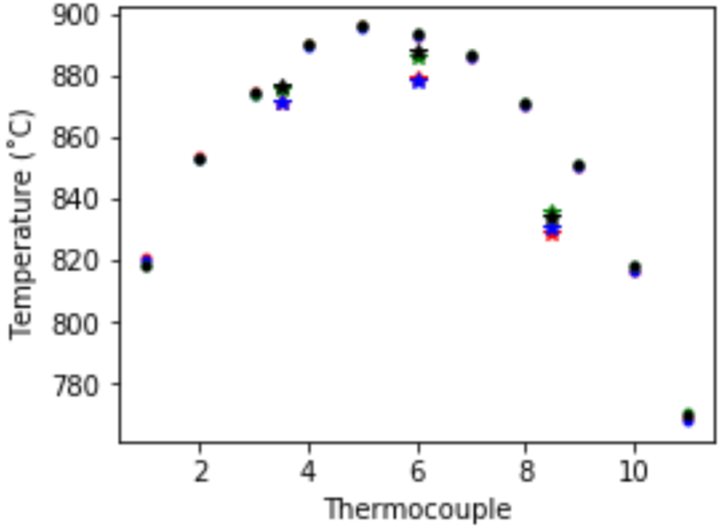
# Temperatures without powder




60 min at 900˚C



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# Tube furnace vs. cone

- Challenge: creating similar conditions to compare
  - Temperature measurements from inert samples?
  - Temperature measurements from PMMA samples?
    - IR camera
    - Thermocouples
  - Suggestions?