

Parameters influencing the mass loss rate in the tube furnace for PMMA

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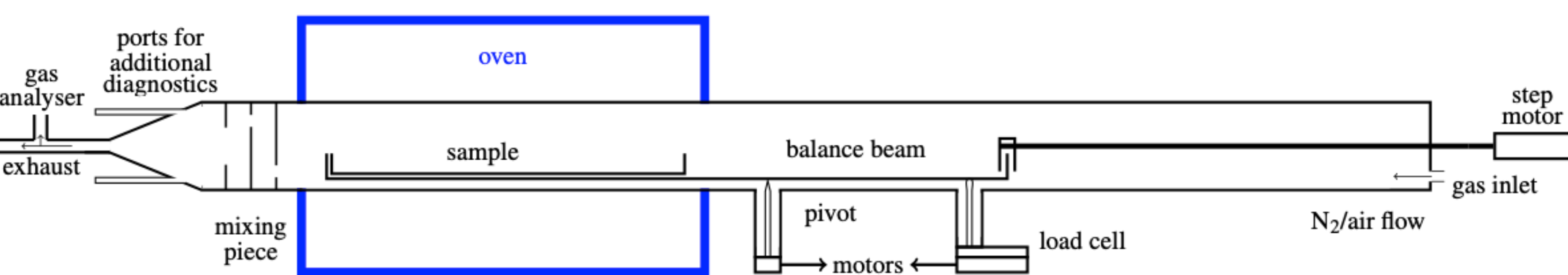
1 Introduction

Classically, the tube furnace as described in ISO 19700 is used for toxicity tests. In this set-up a sample is introduced into a tubular oven at a predefined rate in order to achieve steady state burning. The tube furnace used for this contribution is based on ISO 19700 but has been adapted to enable an online mass loss measurement. The advantage of this set-up is that it allows for gram scale pyrolysis or burning tests under well-defined boundary conditions, compared to other existing gram scale experiments like the cone calorimeter, where a.o. the flow velocities around the sample are not well characterized.

2 Experimental set-up

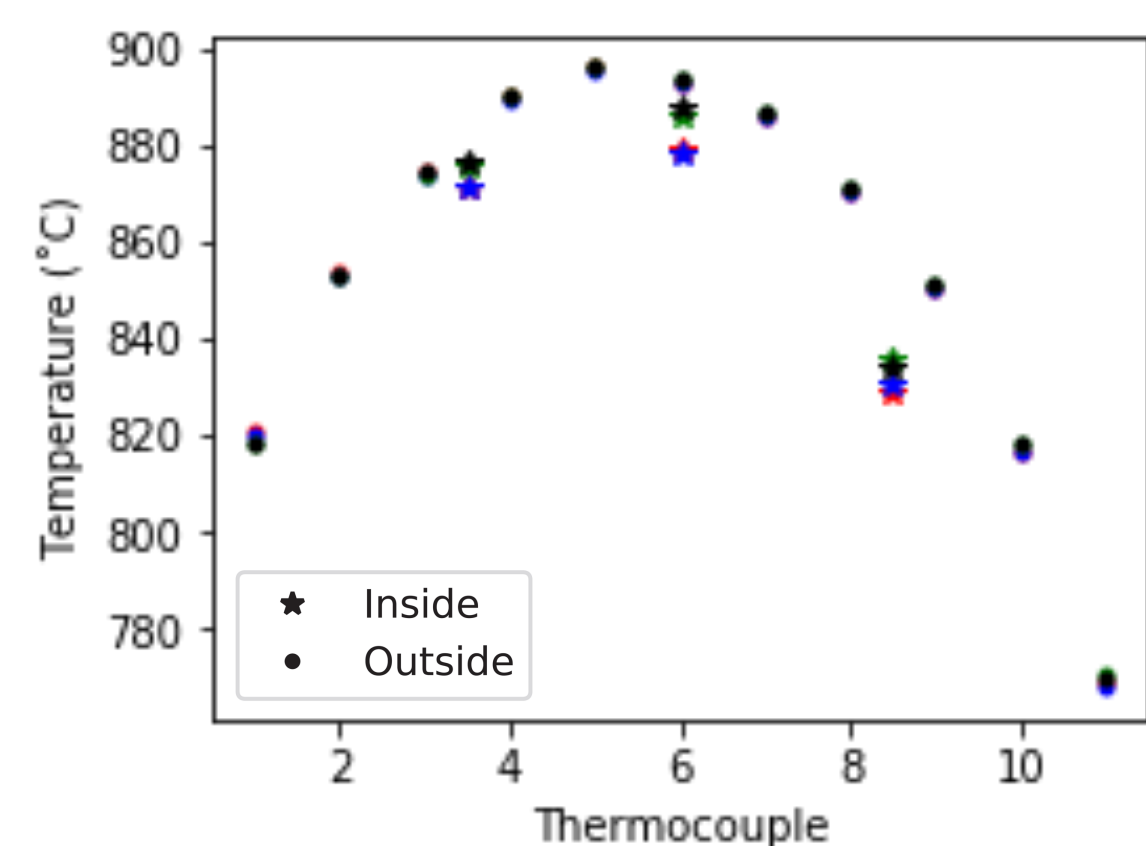
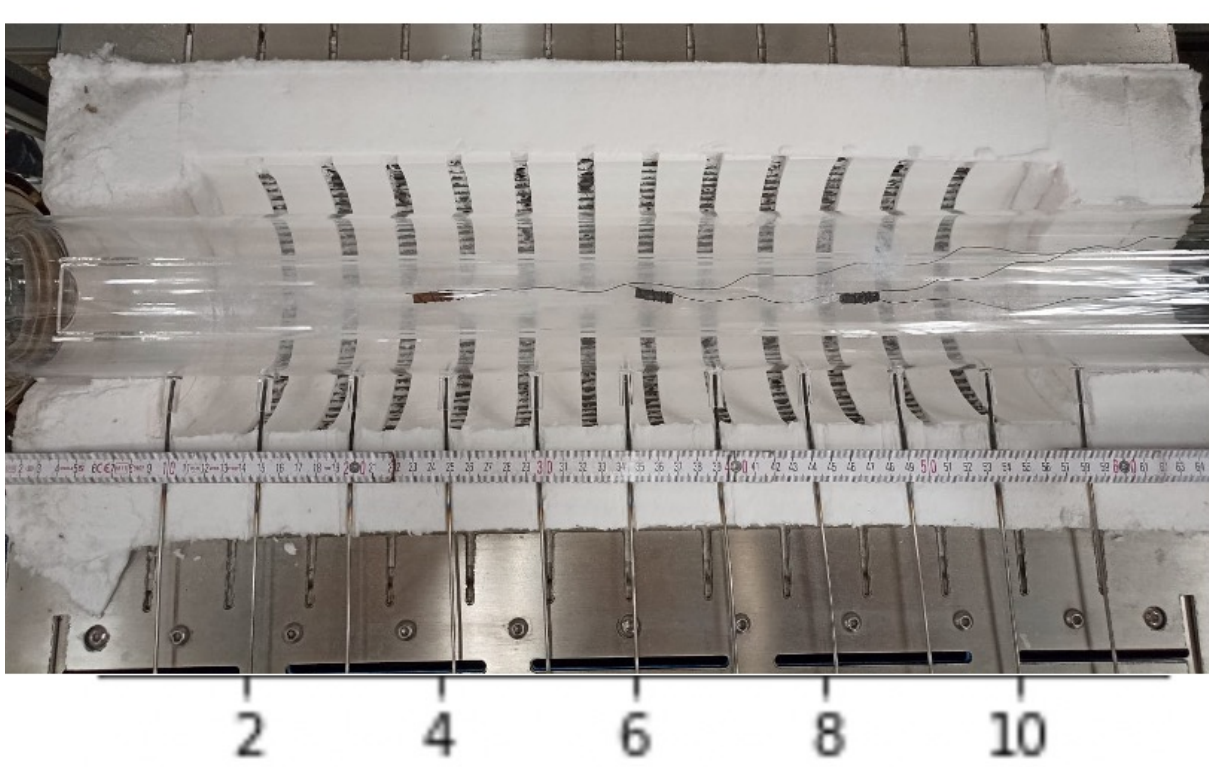
Tube furnace:

- Experiments possible at constant heating rate or at constant temperature
- Heating rate: max. 5 °C/min
- Maximum temperature: 1000 °C
- Atmosphere: adjustable flows and compositions (air or inert)
- Temperature gradient in furnace due to asymmetric flow conditions



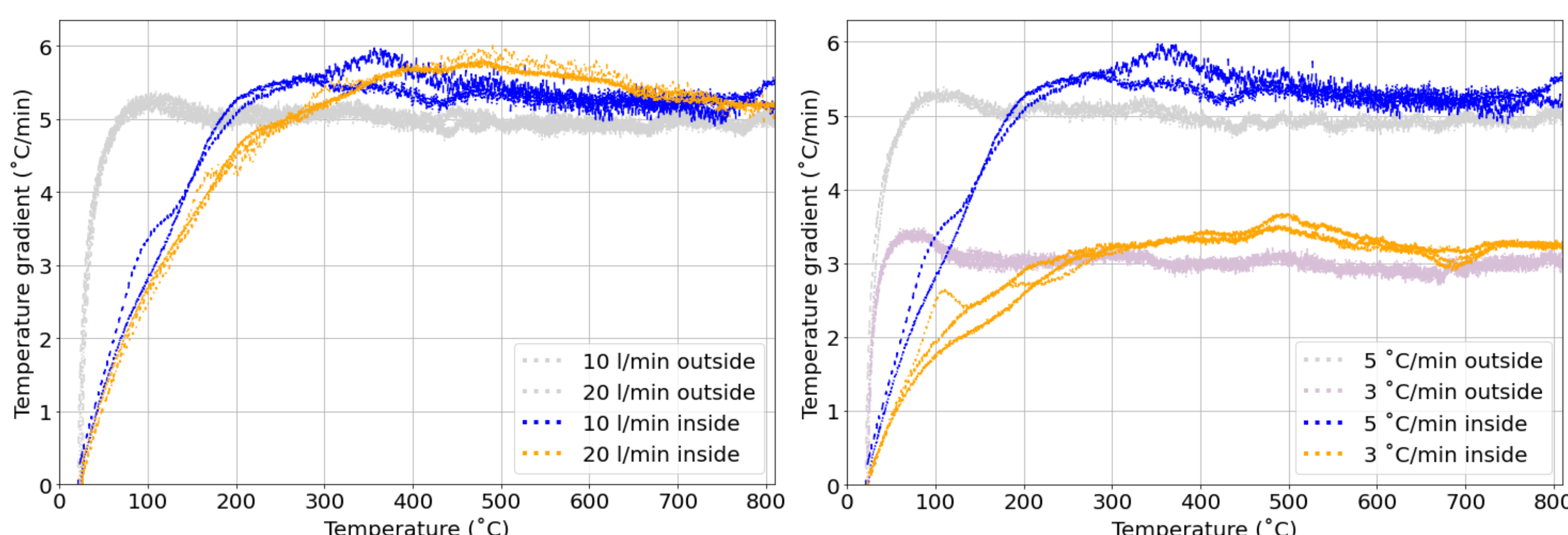
Measurements:

- Gas analyzer: CO, CO₂ and O₂
- Cantilever balance: online mass loss measurement in the center of mass of the sample
- Thermocouples at the in-and outside of the tube



Heating rate:

- Controlled by changing the oven power over time
- Manually optimized for defined heating rates
- Optimized using outside thermocouples
- Above 200 °C: variation in heating rate < 1 °C/min



3 Experimental Results

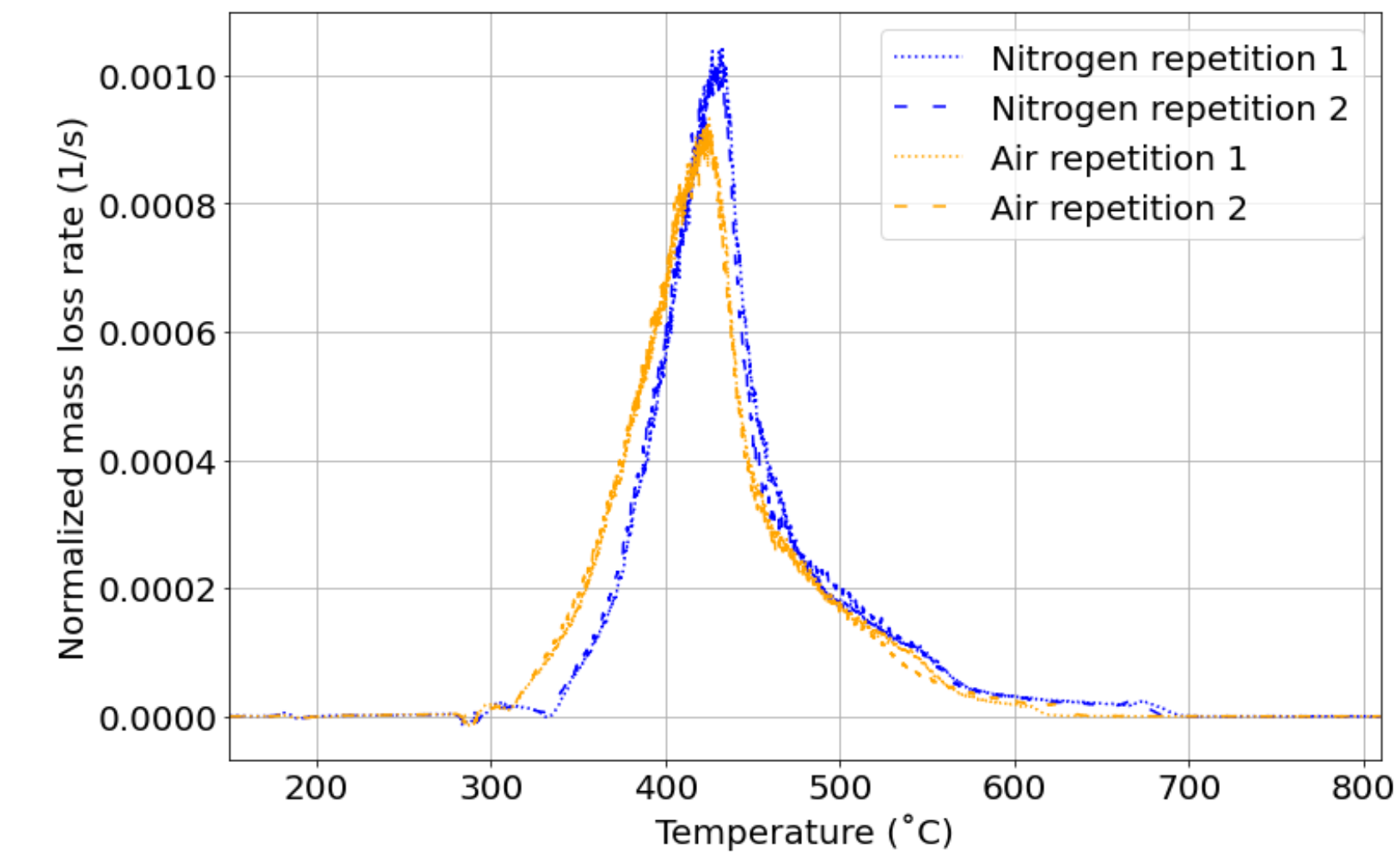
Material: cast black PMMA (9H10 by Evonik)

Dimensions: 500 mm x 20 mm x thickness

Heating rate: 5 °C/min

Atmosphere:

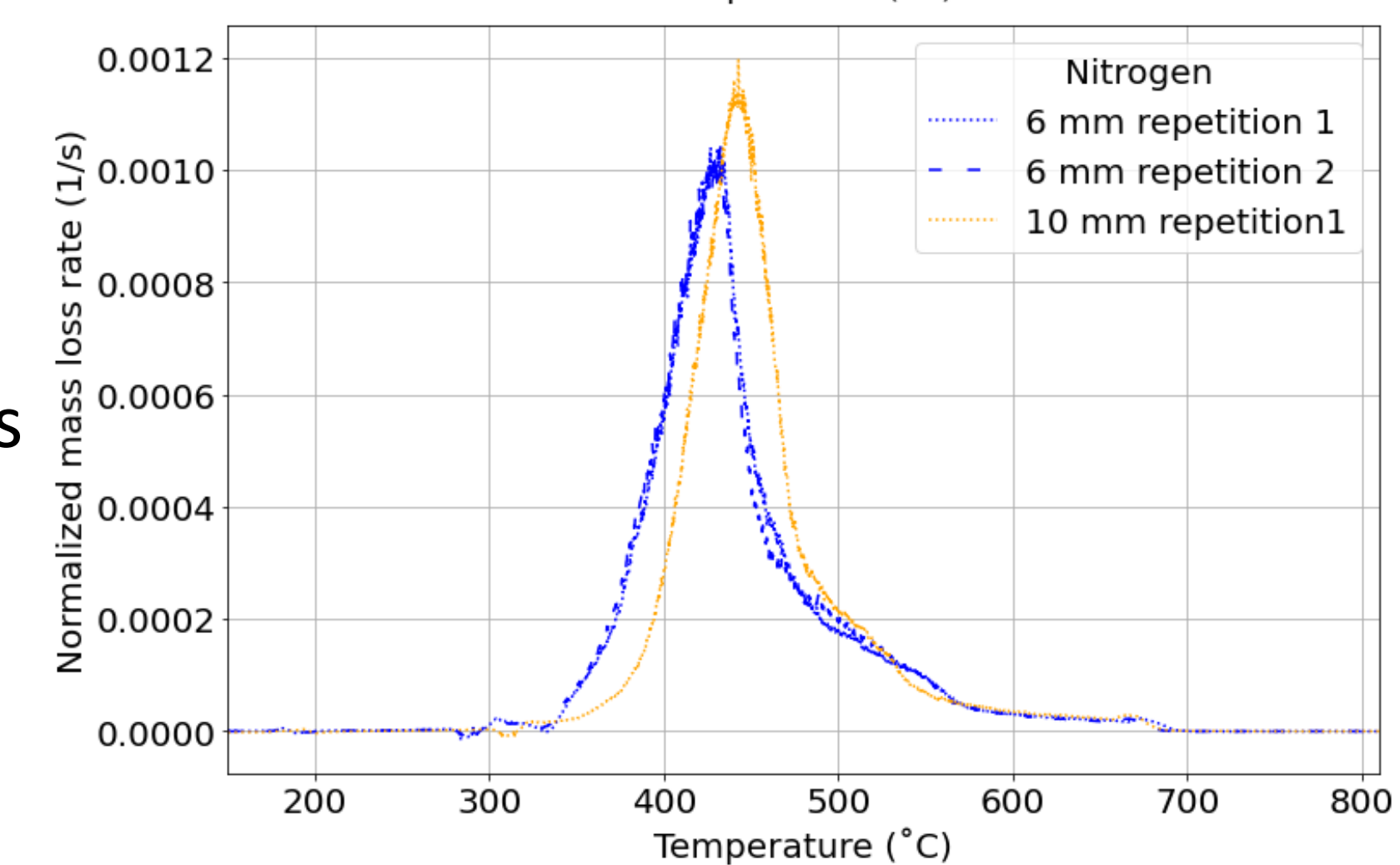
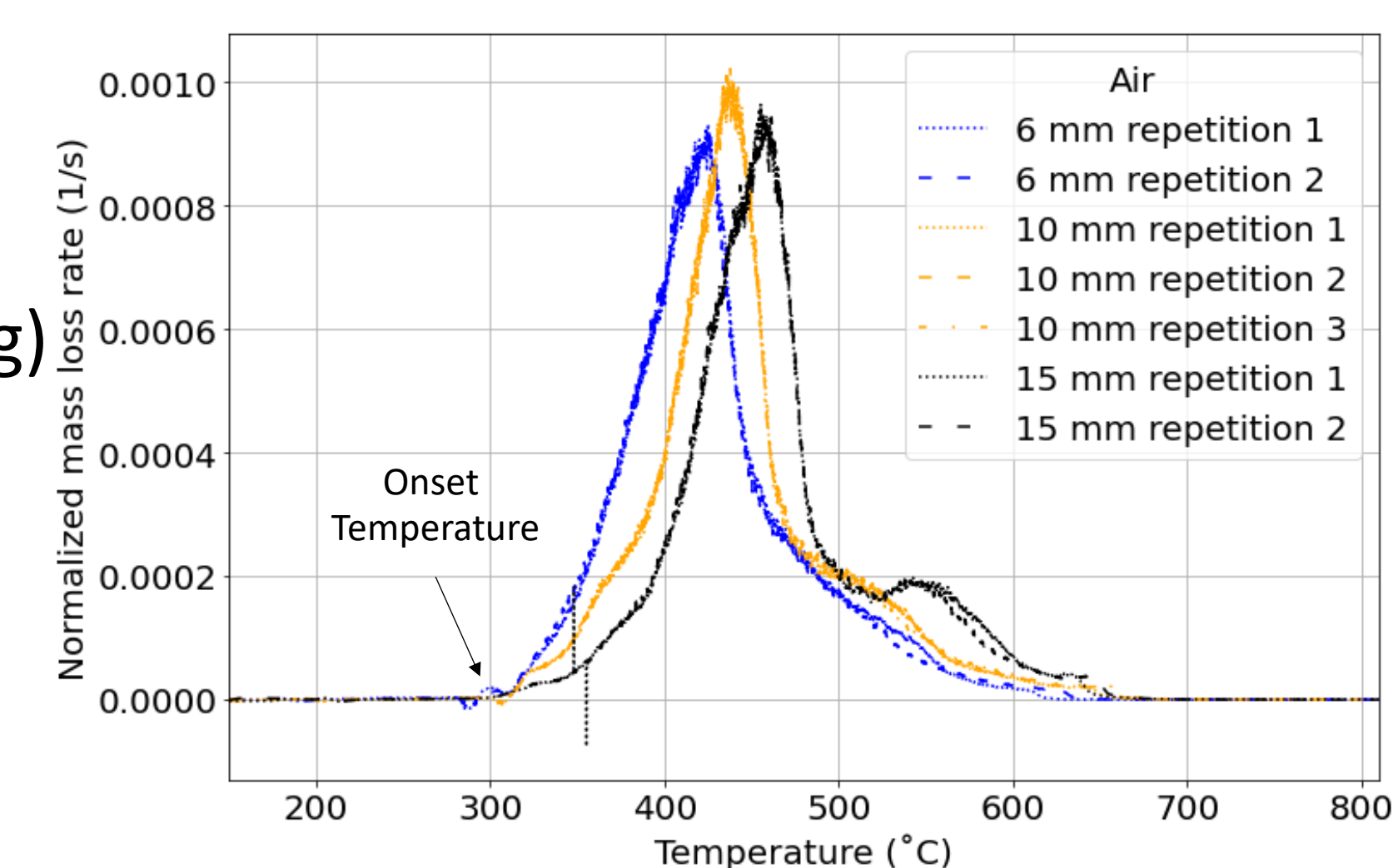
- N₂ atmosphere, air atmosphere
- Reaction under air atmosphere starts ~20 °C before reaction under N₂ atmosphere; similar as in the TGA [1].



Sample weight:

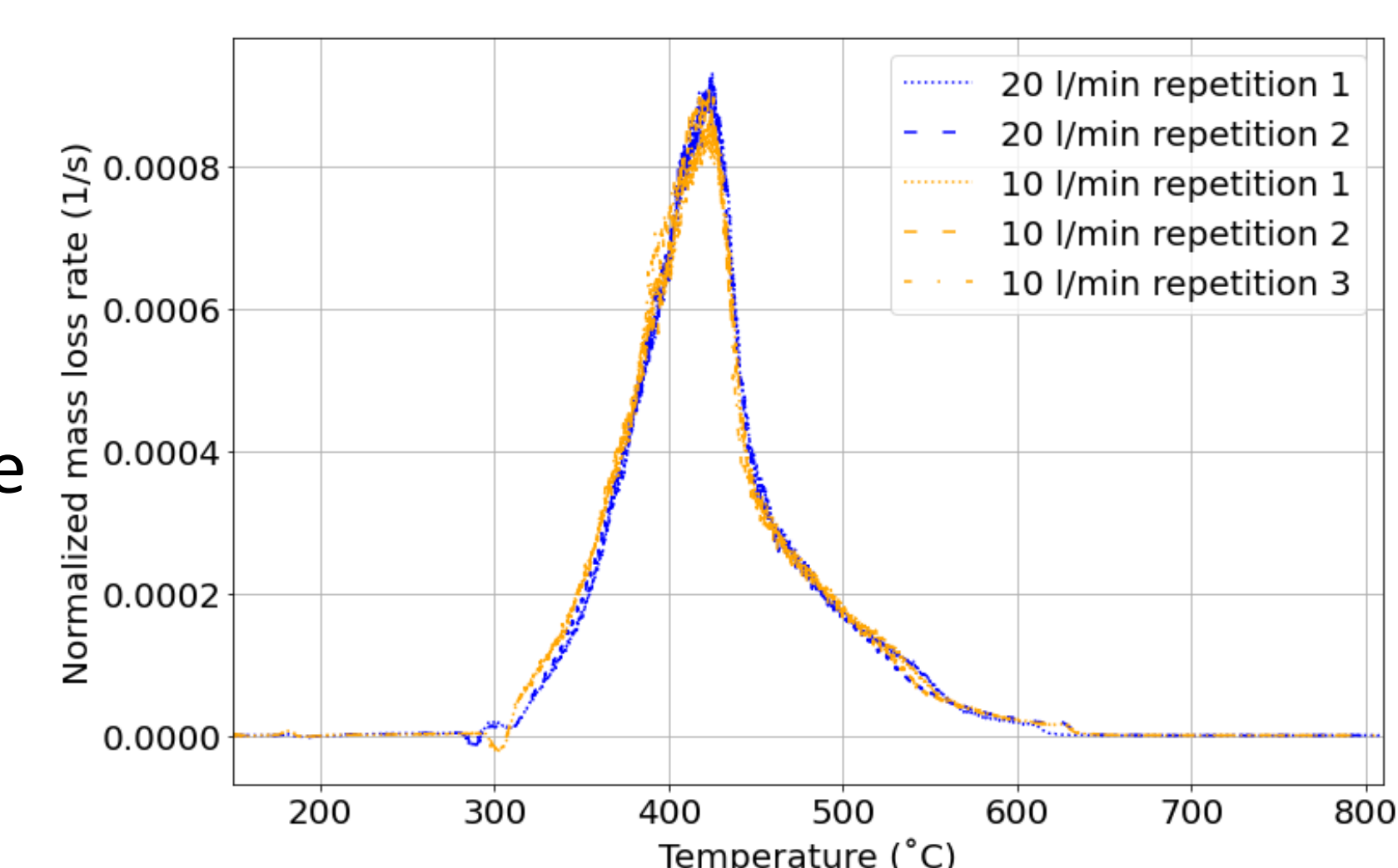
- 4 different sample thicknesses: 6 mm (~69 g), 10 mm (~113 g), 15 mm (~163 g), 20 mm (~222 g)
- Spontaneous ignition for:
 - 10 l/min: 10 mm, 15 mm, 20 mm
 - 20 l/min: 20 mm

- Good agreement between repetition experiments
- Reaction onset remains similar for different masses
- Main peak shifts to higher temperature for thicker samples due to the thermal lag



Flow rate:

- 10 l/min and 20 l/min
- Atmosphere: air
- Sample thickness: 6 mm
- Flow rate does not influence the mass loss rate, unless a lower flow rate results in ignition



4 Outlook

- Flow and sample mass investigation for N₂ atmosphere: No ignition problem for thick samples
- Conduct experiments at different heating rates: Is the effect of a lower heating rate similar as the effect of a lower mass?
- Experiments with temperature measurements: What is the influence of the temperature gradient in the sample as well as the effect of the non-homogeneous surface temperature on the mass loss rate?

[1] Fateh, T., et al., Experimental and modelling studies on the kinetics and mechanisms of thermal degradation of polymethyl methacrylate in nitrogen and air, Journal of analytical and applied pyrolysis, 120:423-433, 2016.



This work is part of the BESKID project, which is funded by the German Federal Ministry of Education and Research: "Research for Civil Safety 2018 - 2023" www.sifo.de

Funding for the topic: "Artificial Intelligence in Civil Safety Research II"
Project name: Design fire simulations in rail vehicles using AI-based data (BESKID)