



# Particles-heated sulphuric acid decomposition reactors

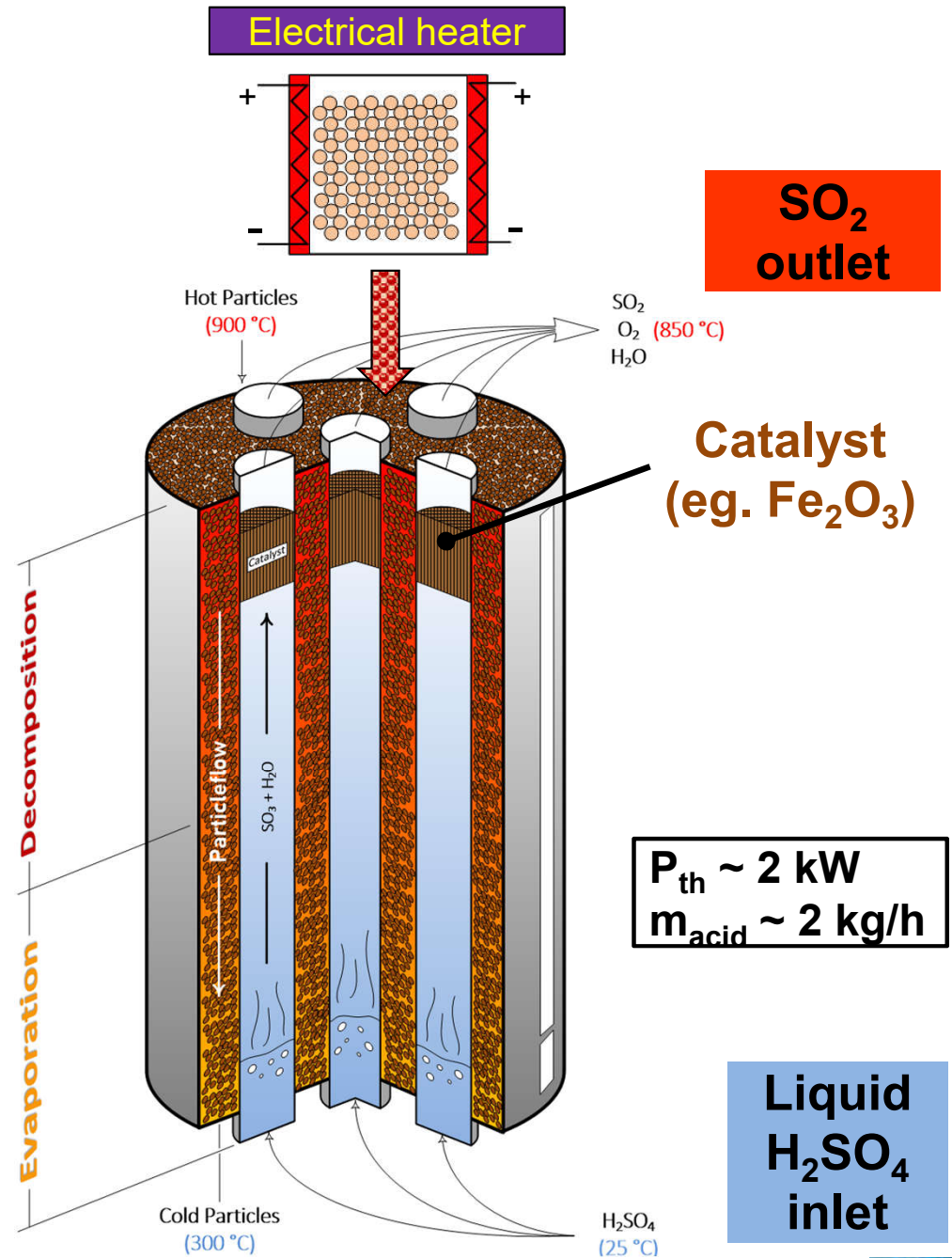
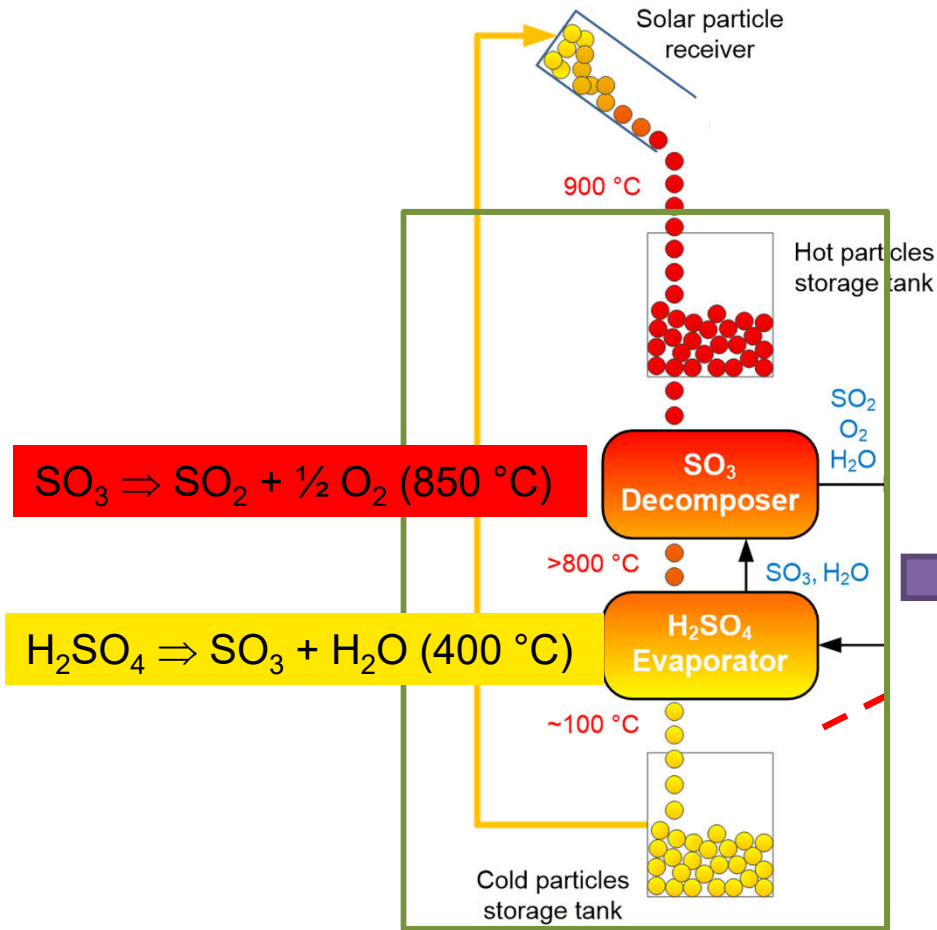
## PEGASUS FINAL WORKSHOP – 09.09.2021

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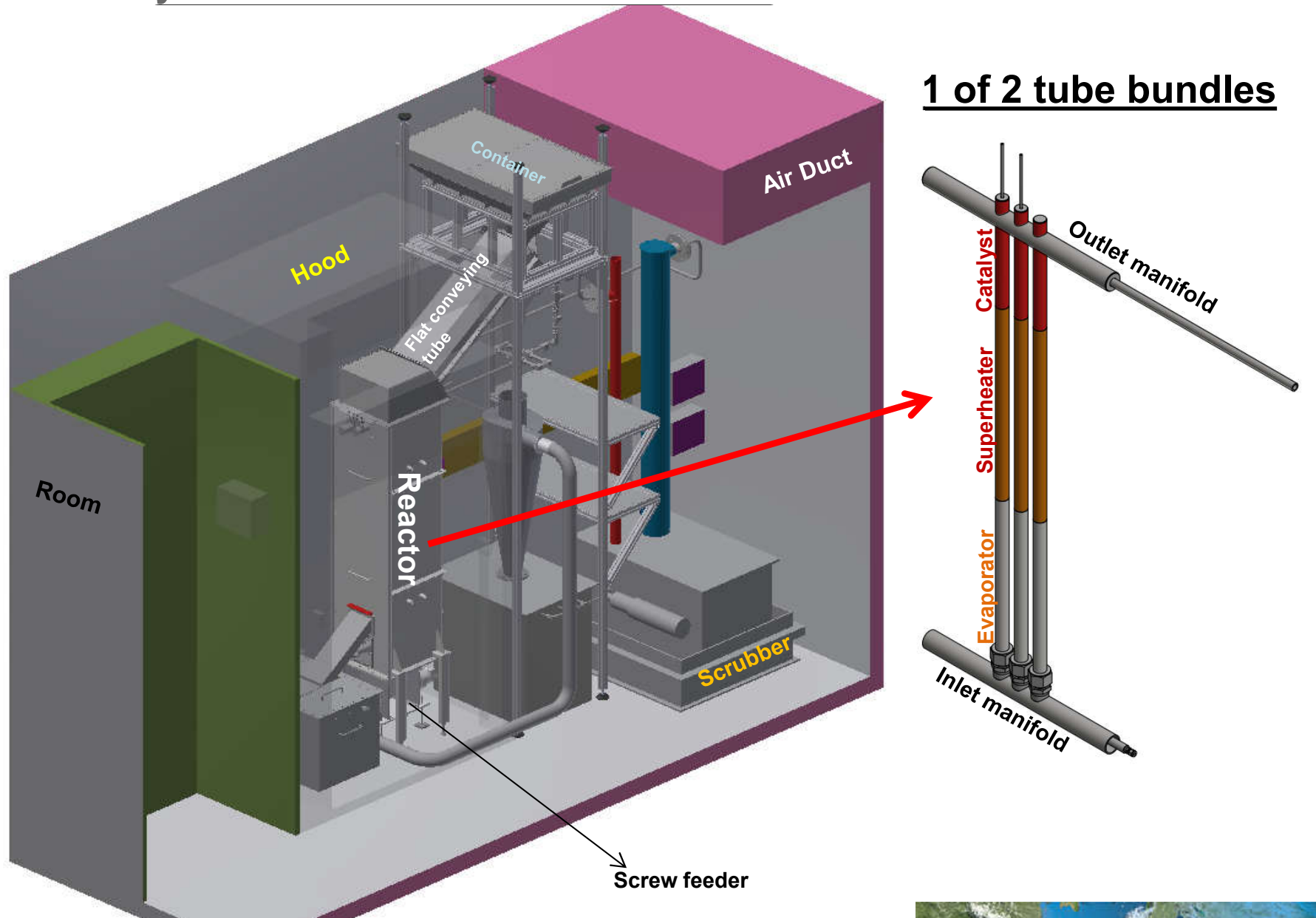


# PEGASUS Process diagram



# Particles-heated decomposition reactor - CAD

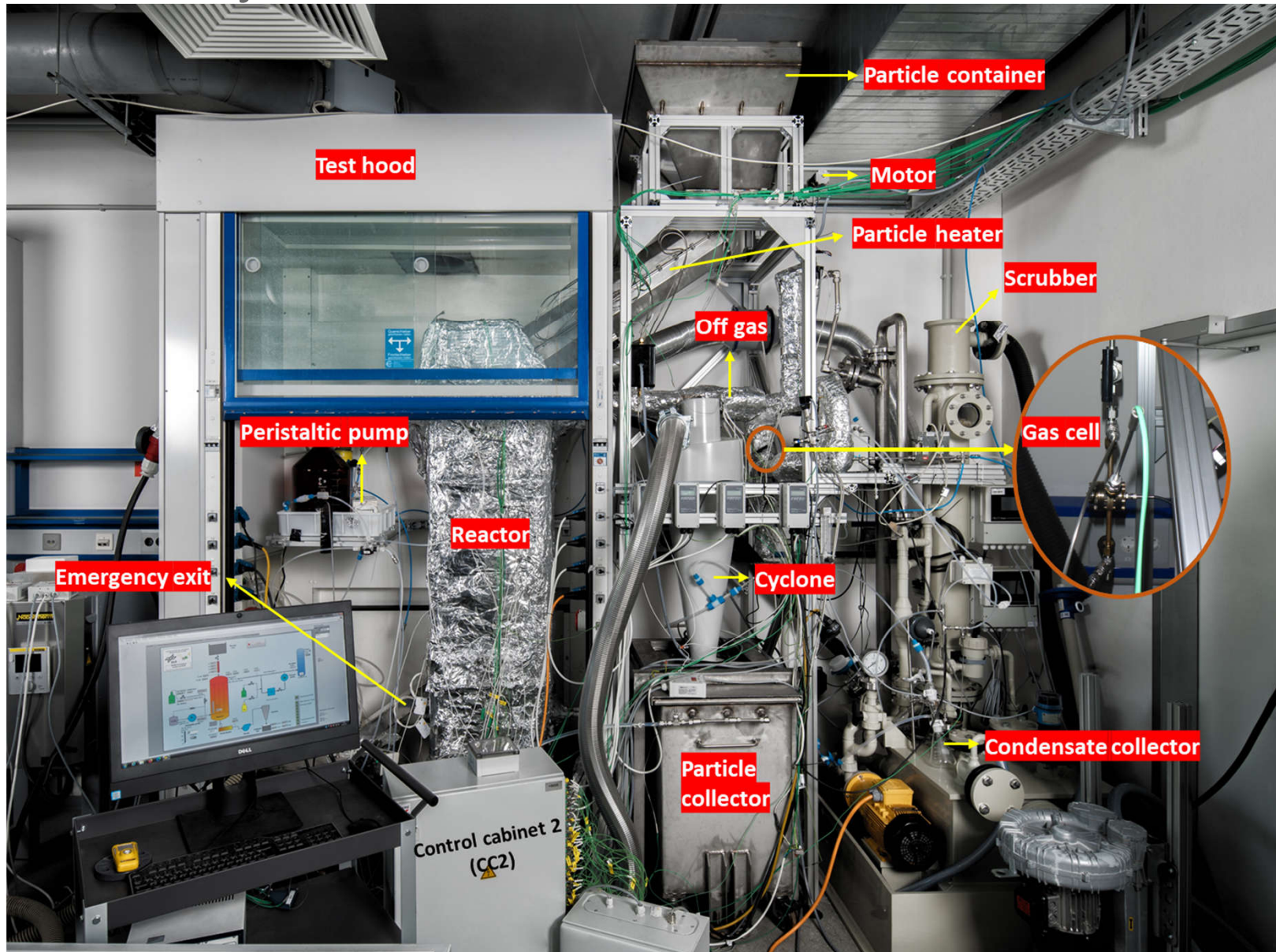
## Overview of system at DLR lab in Juelich





# Particles-heated decomposition reactor - Assembly

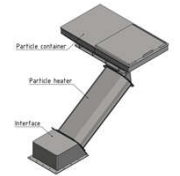
## Overview of system at DLR lab in Juelich



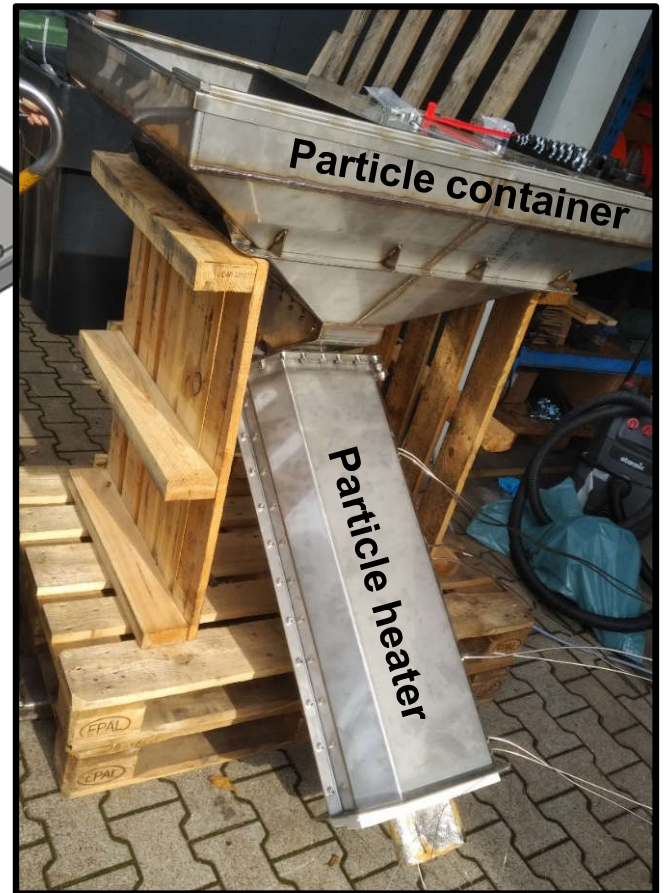
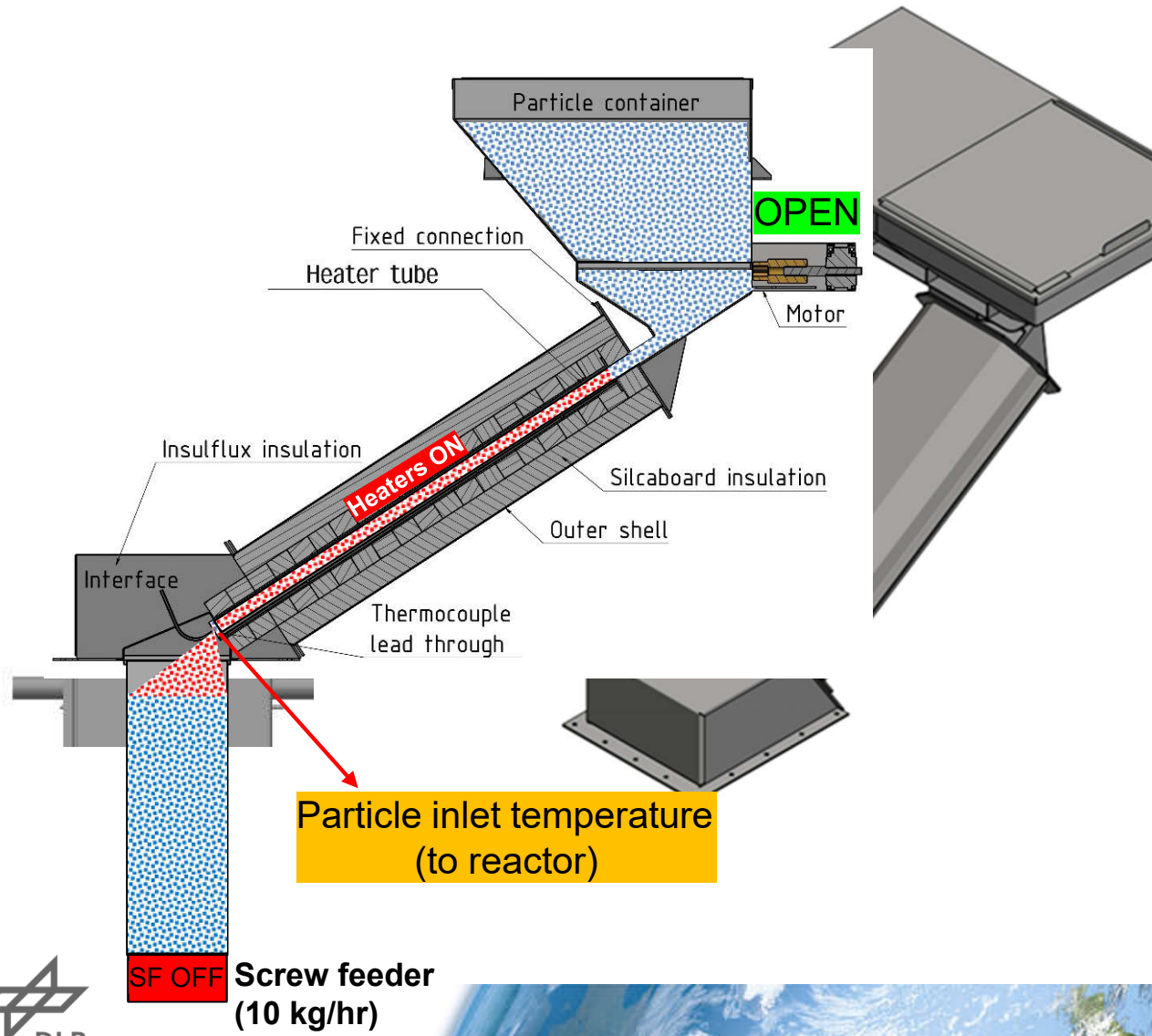


# Particles-heated decomposition reactor

## Development of new Particle Heater

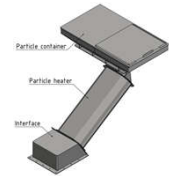


### Cross-section view



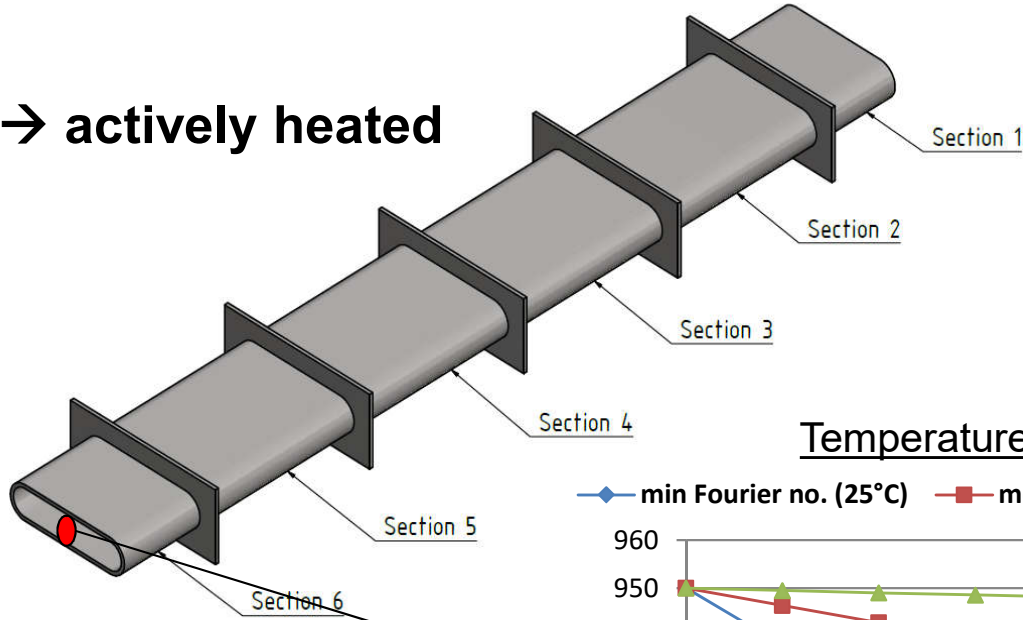
# Particles-heated decomposition reactor

## Particle Heater – Thermodynamic model



Flat conveying tube

S2-S5 → actively heated

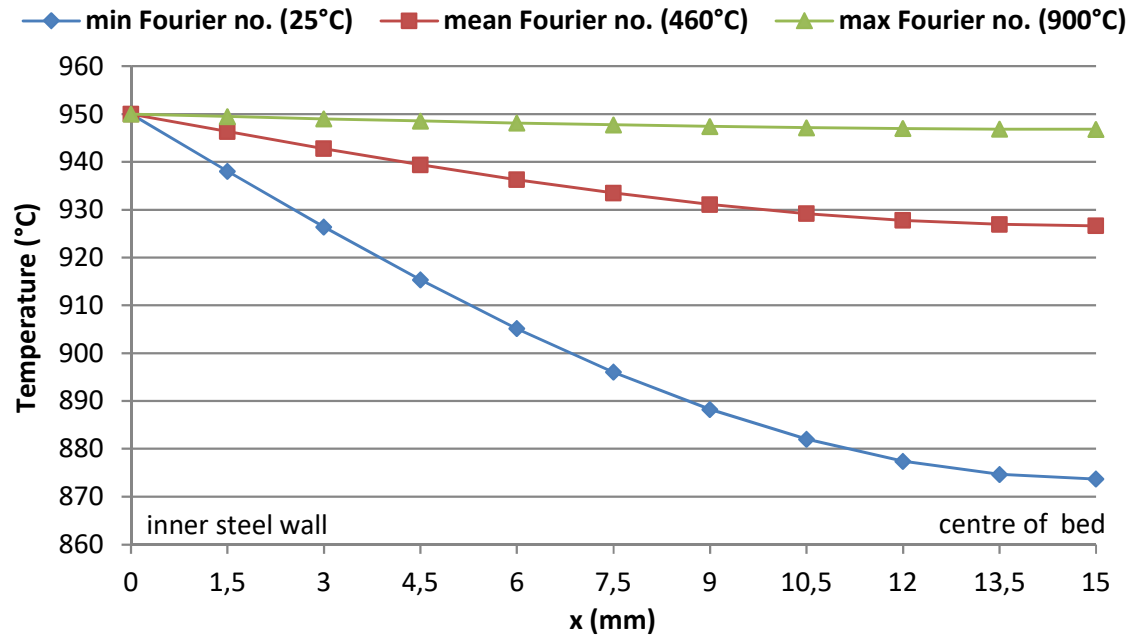


Result:

$t = \text{approx. } 50 \text{ min}$   
 → Core temperature  $\geq 870 \text{ }^\circ\text{C}$

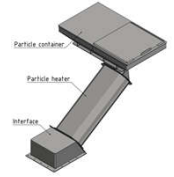
→ Ribs removed

Temperature profile in tube cross-section

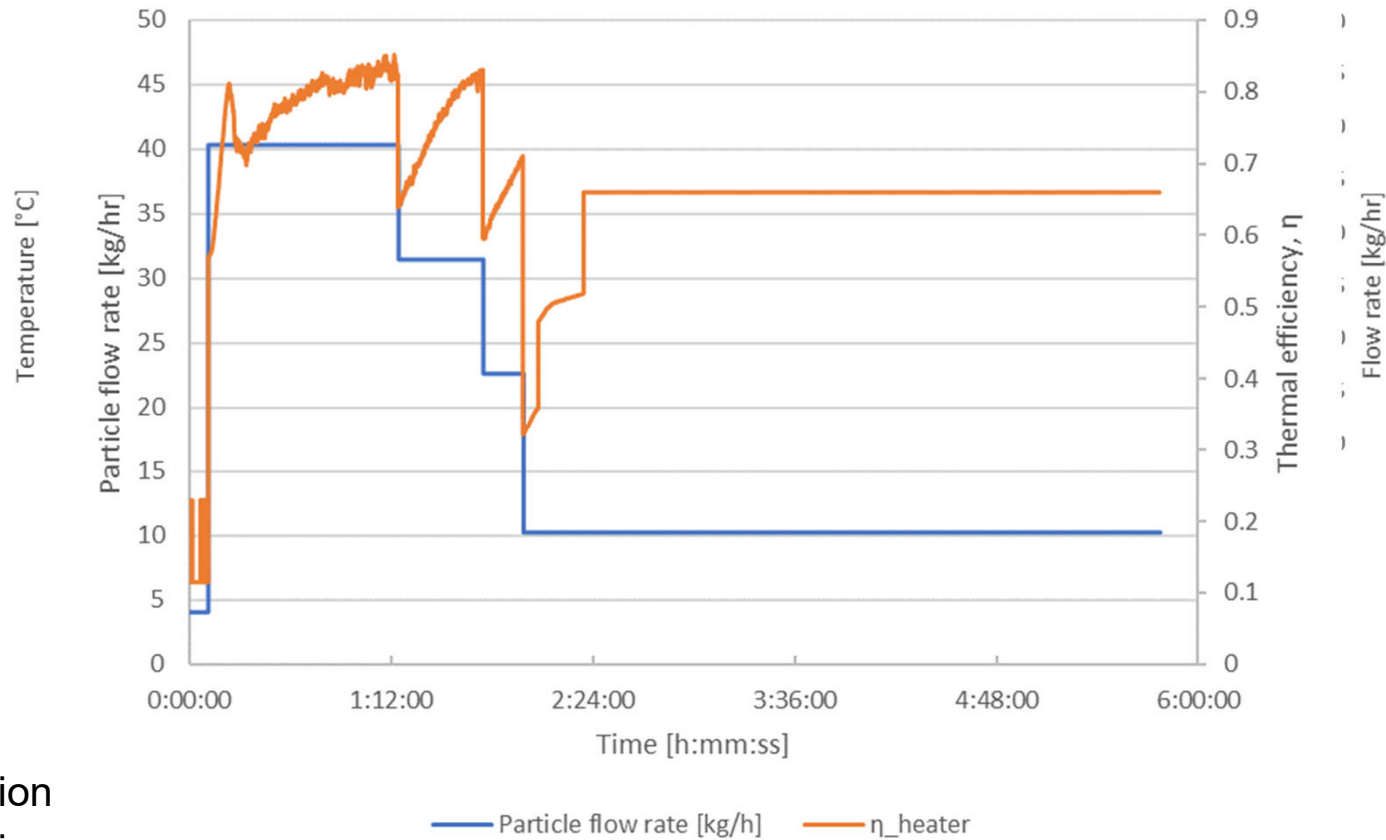


# Particles-heated decomposition reactor

## Particle Heater – Testing



### Heater performance



H1: Heater 1 (section

H2: Heater 2 (section

H3: Heater 3 (section 4)

H4: Heater 4 (section 5)

→ The particle bed has reached more than 870°C as calculated

→ At 10 kg/hr particle flow rate, the efficiency was 65% (steady state)



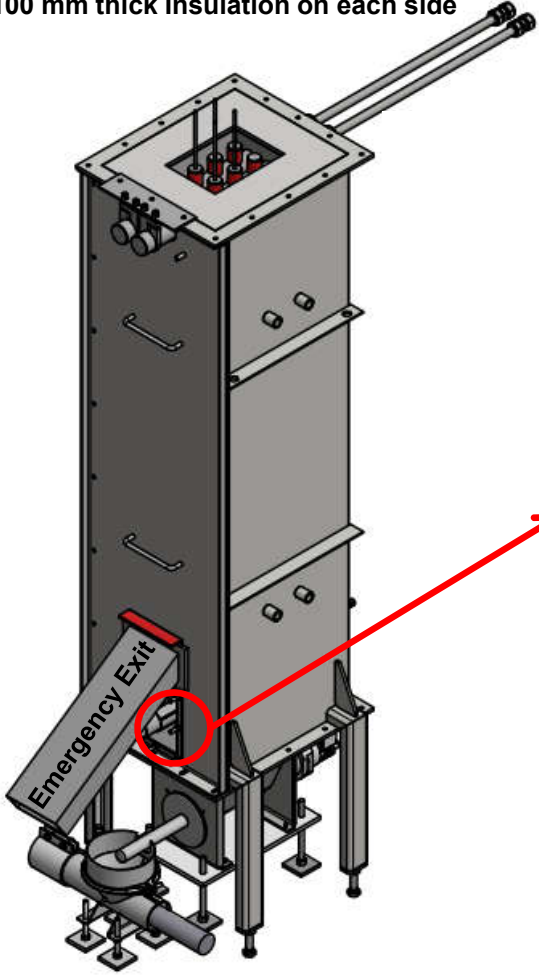


# Particles-heated decomposition reactor

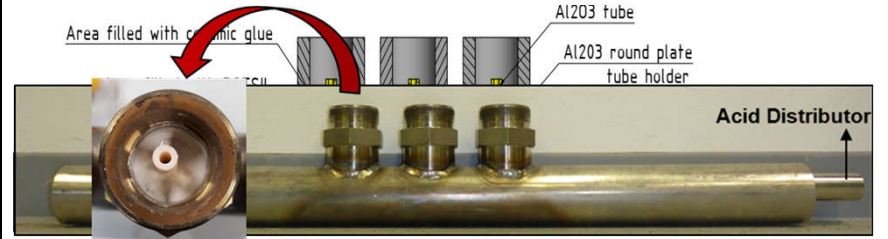
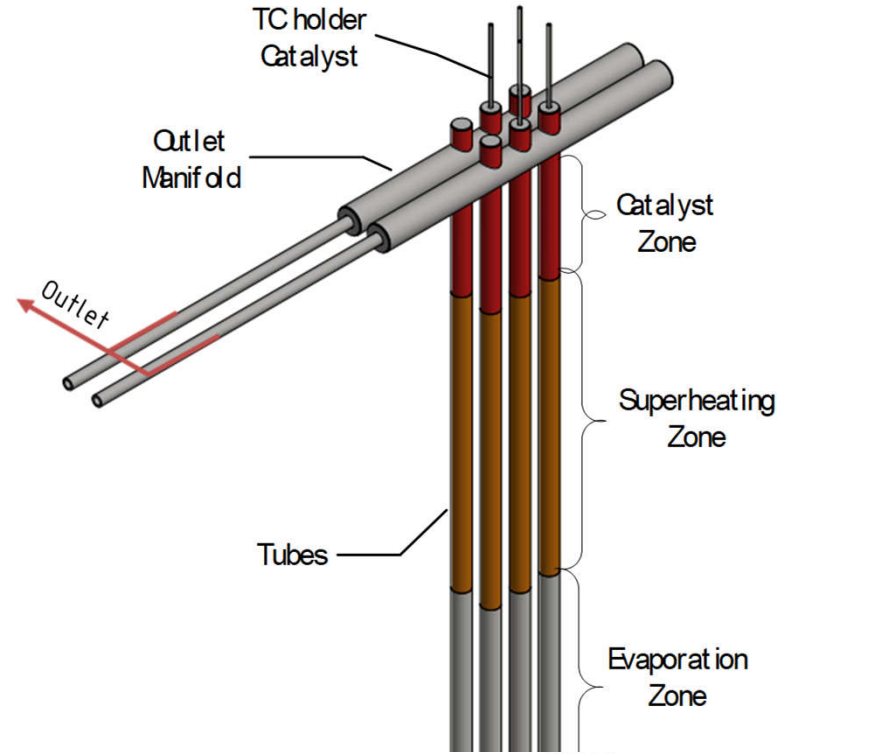
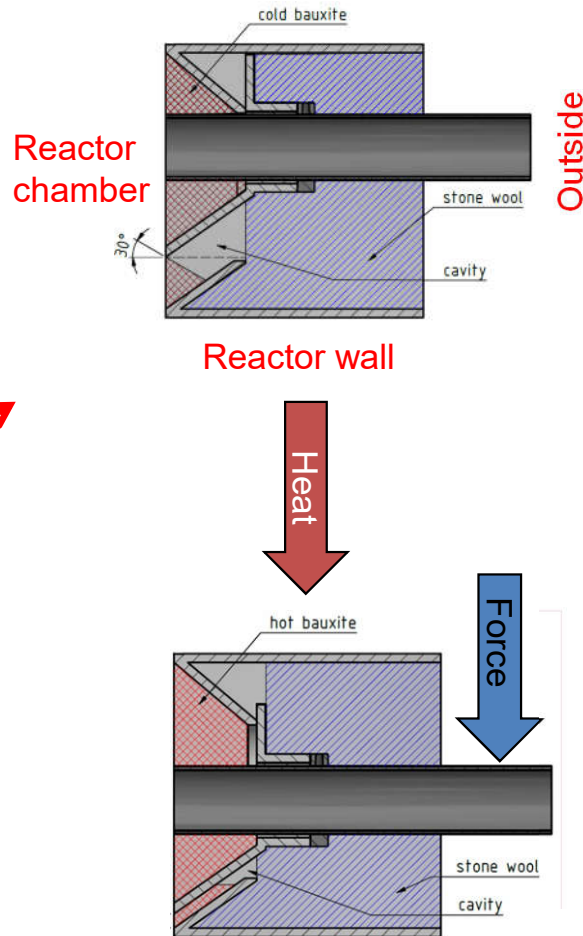
## Reactor shell and tubes



100 mm thick Insulation on each side



### Compensation of thermal expansion

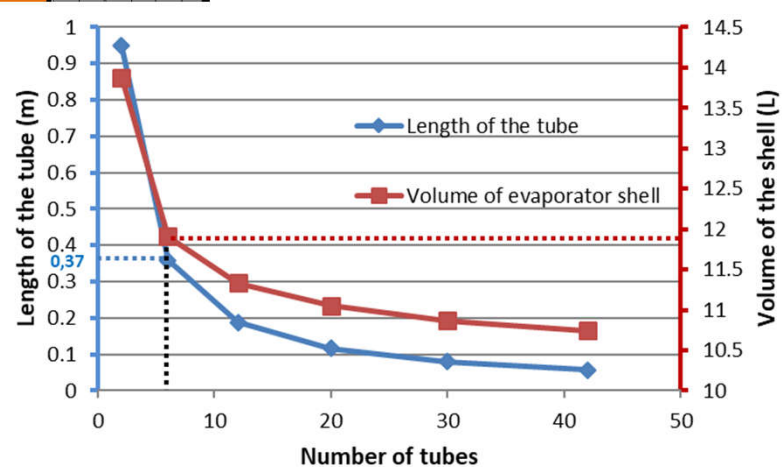
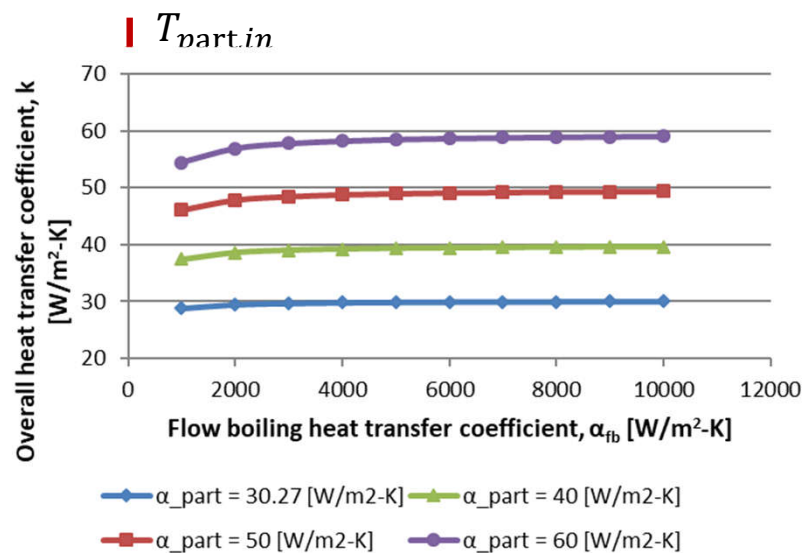
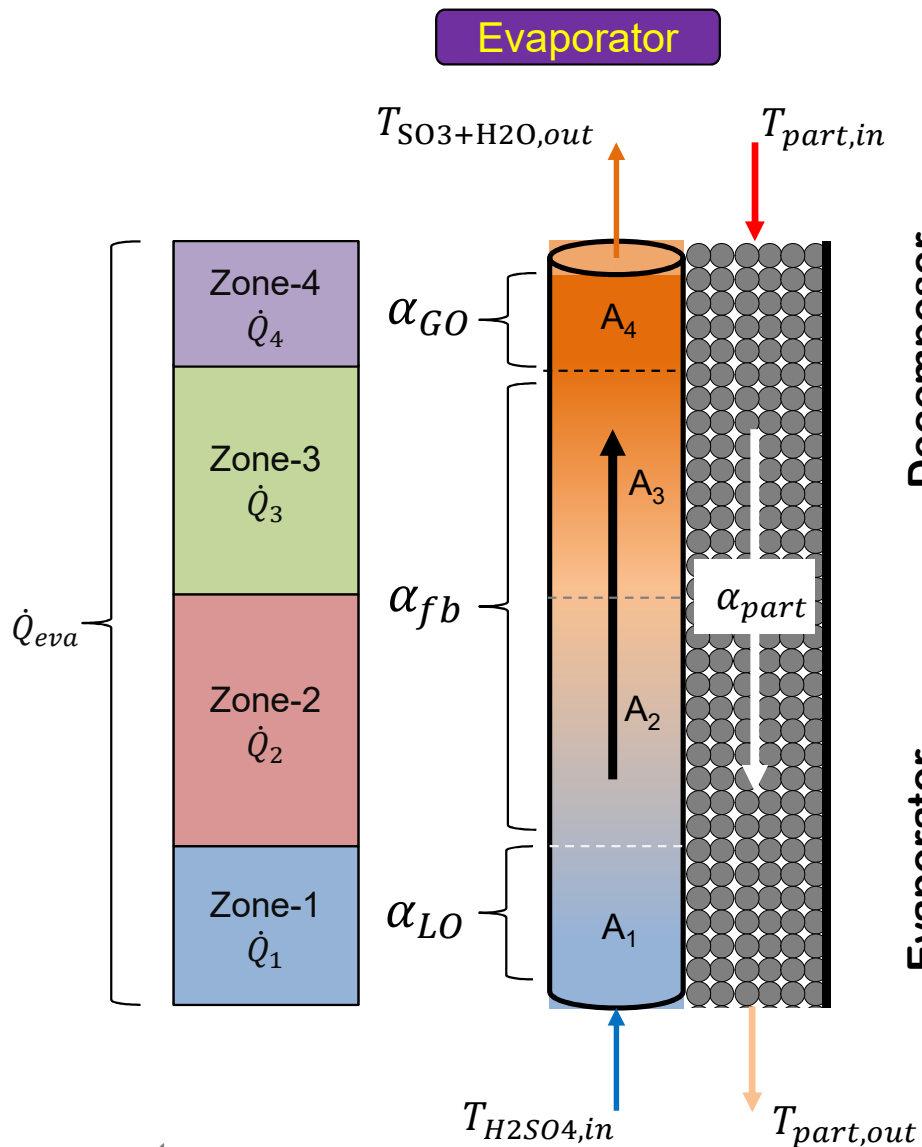
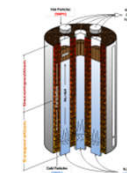


Corrosion resistant acid distributor made of Hastelloy and ceramic compounds

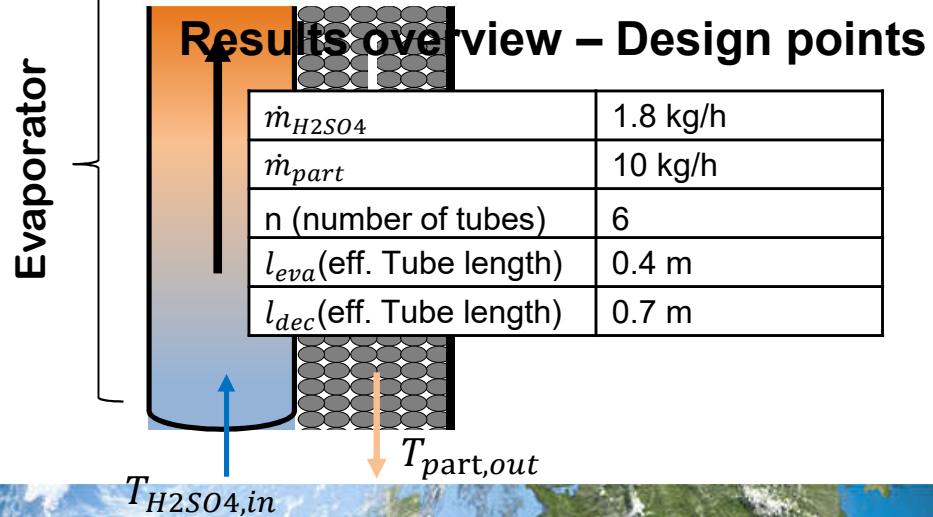
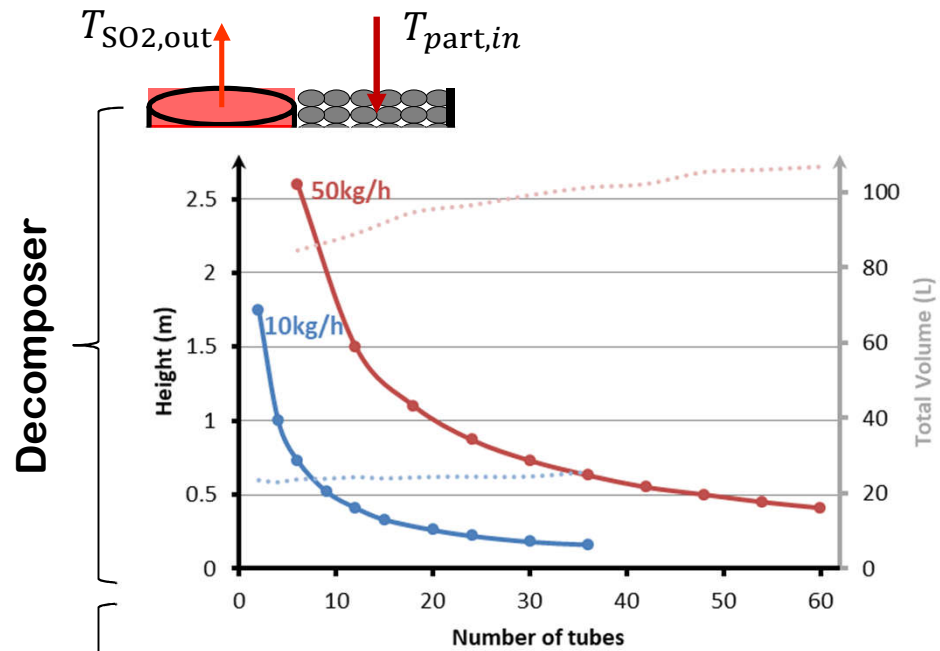
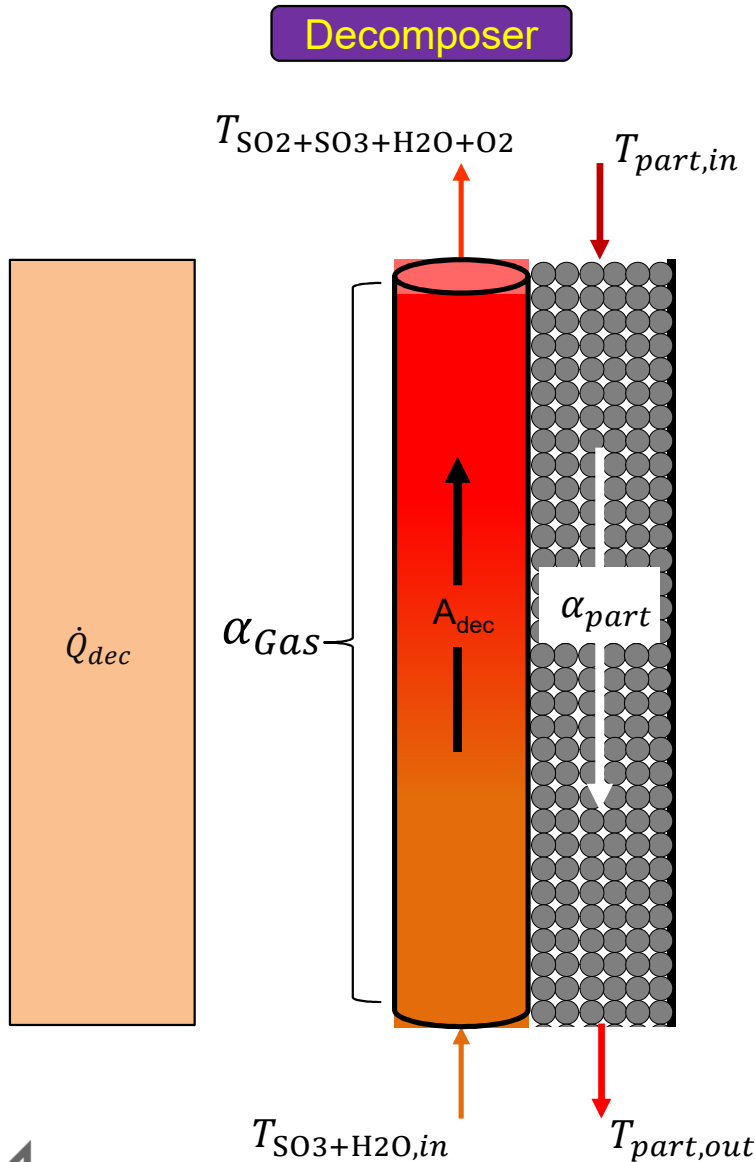
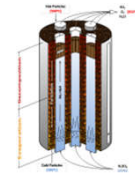




# Particles-heated decomposition reactor Sizing



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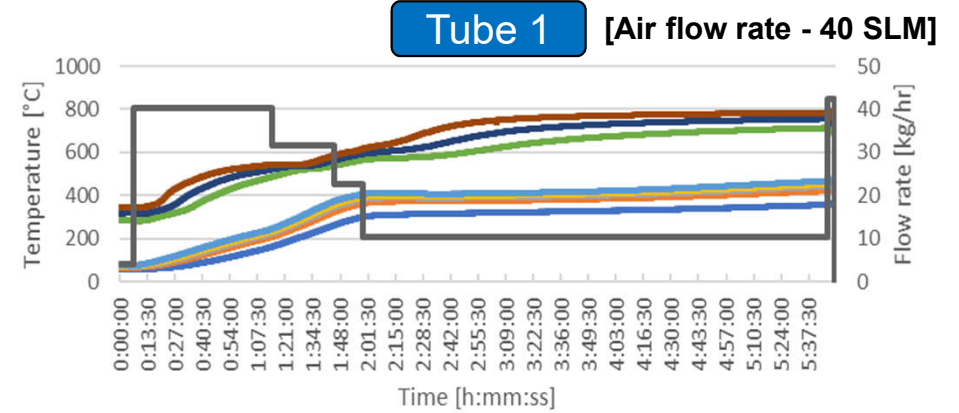
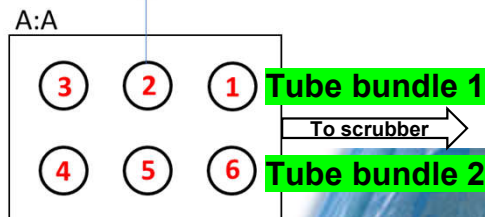
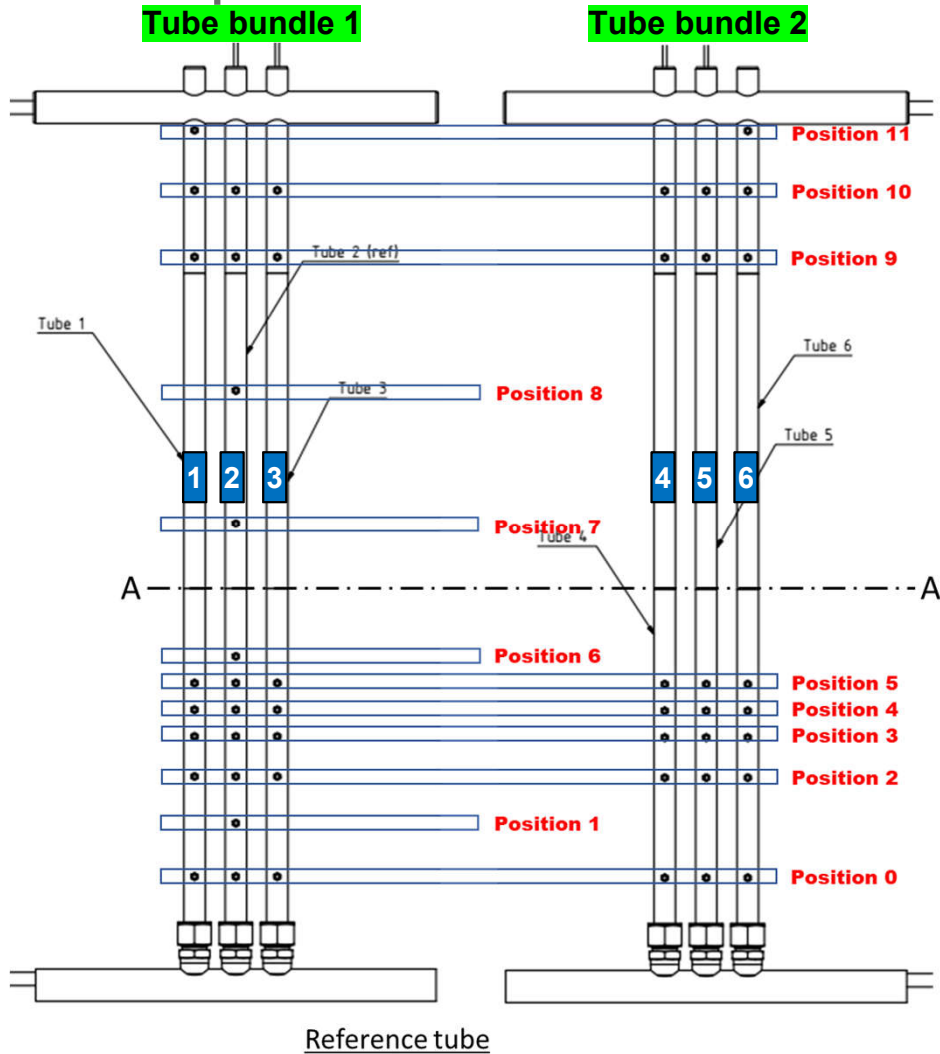
## Results overview – Design points

$\dot{m}_{H_2SO_4}$	1.8 kg/h
$\dot{m}_{part}$	10 kg/h
n (number of tubes)	6
$l_{eva}$ (eff. Tube length)	0.4 m
$l_{dec}$ (eff. Tube length)	0.7 m

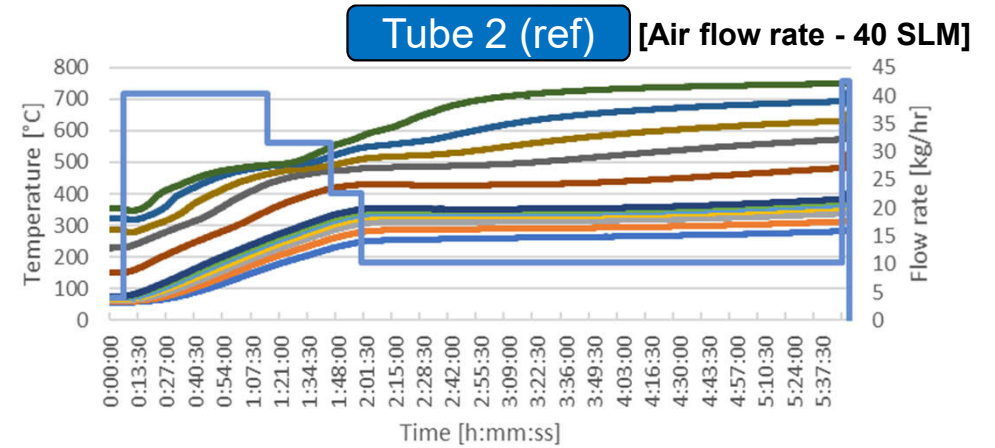


# Particles-heated decomposition reactor

## Temperature distribution – Thermal test (28.05.2021)



- TR113 [°C] Pos. 0      TR114 [°C] Pos. 2      TR115 [°C] Pos. 3
- TR116 [°C] Pos. 4      TR117 [°C] Pos. 5      TR118 [°C] Pos. 9
- TR119 [°C] Pos. 10    TR120 [°C] Pos. 11      Particle flow rate [kg/h]

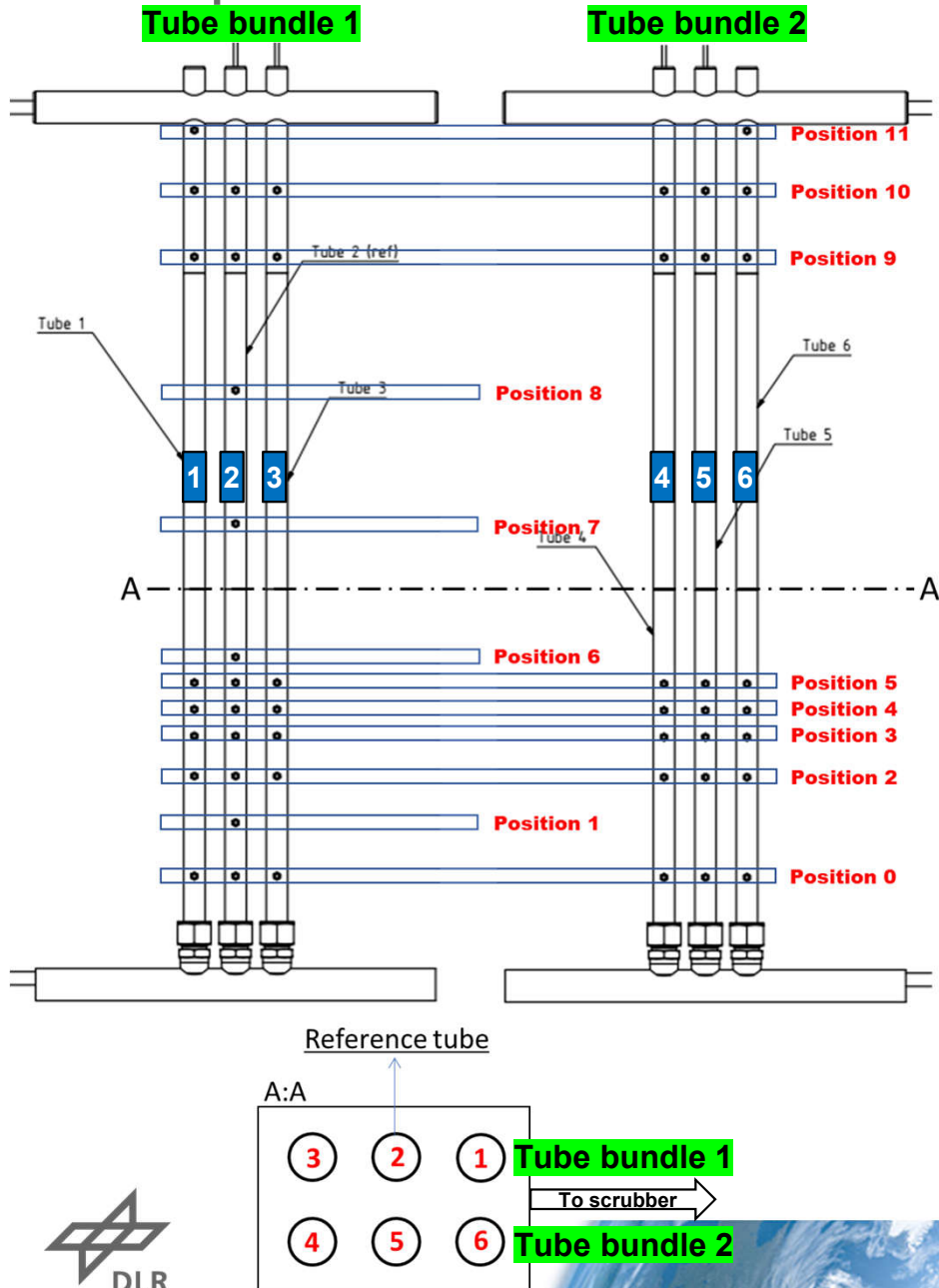


- TR101 [°C] Pos. 0      TR102 [°C] Pos. 1      TR103 [°C] Pos. 2
- TR104 [°C] Pos. 3      TR105 [°C] Pos. 4      TR106 [°C] Pos. 5
- TR107 [°C] Pos. 6      TR108 [°C] Pos. 7      TR109 [°C] Pos. 8
- TR110 [°C] Pos. 9      TR111 [°C] Pos. 10      TR112 [°C] Pos. 11
- Particle flow rate [kg/h]

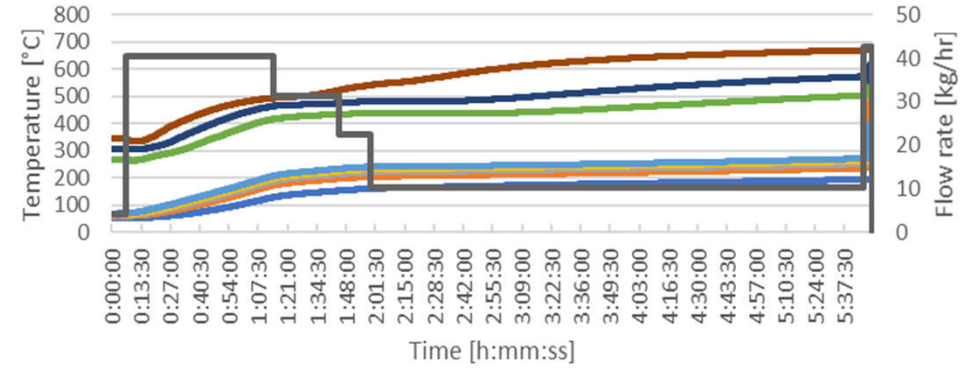


# Particles-heated decomposition reactor

## Temperature distribution – Thermal test (28.05.2021)

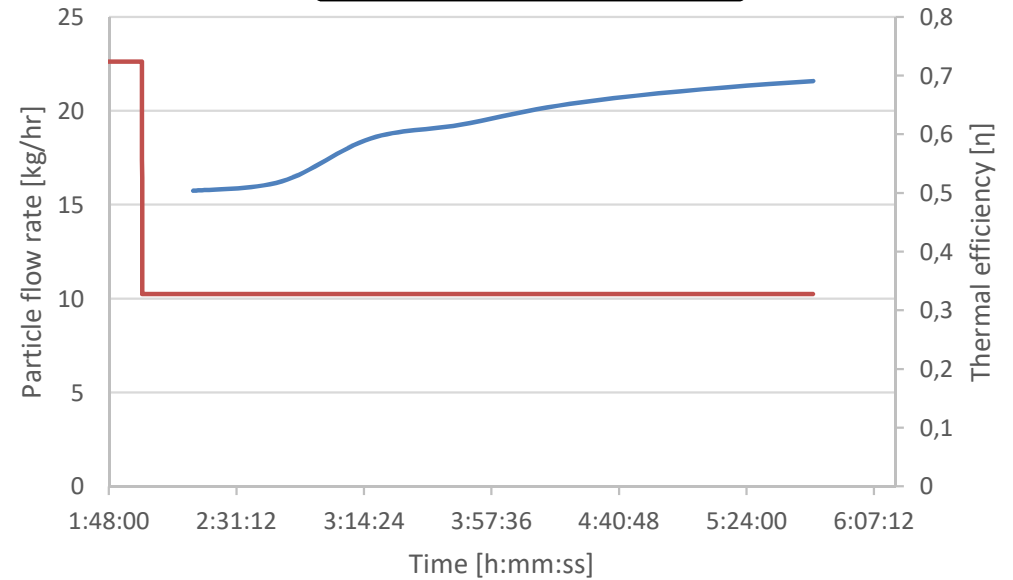


**Tube 3** [Air flow rate - 40 SLM]



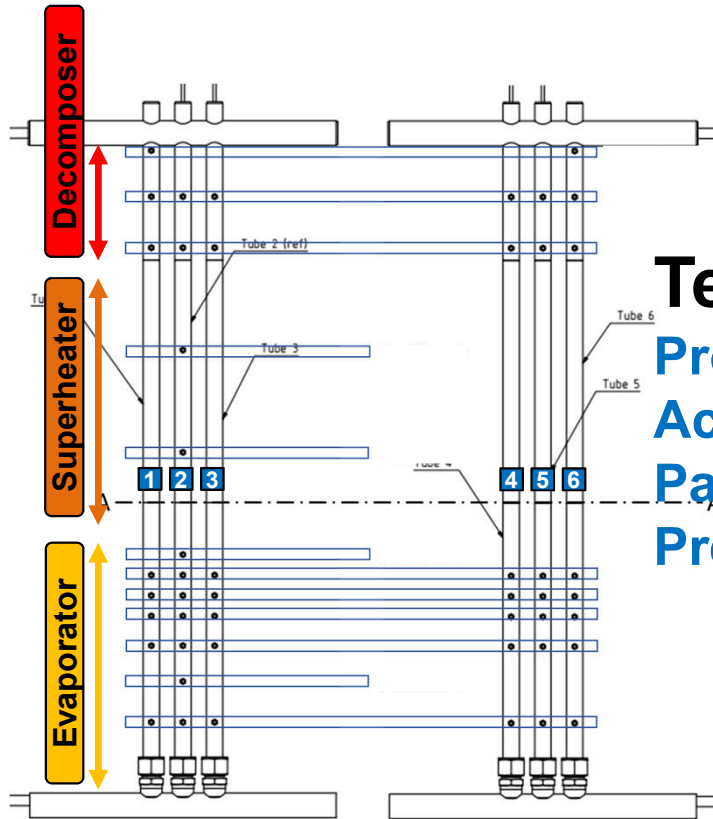
- TR121 [°C] **Pos. 0**
- TR122 [°C] **Pos. 2**
- TR123 [°C] **Pos. 3**
- TR124 [°C] **Pos. 4**
- TR125 [°C] **Pos. 5**
- TR126 [°C] **Pos. 9**
- TR127 [°C] **Pos. 10**
- TR128 [°C] **Pos. 11**
- Particle flow rate [kg/h]

**Thermal efficiency,  $\eta$**



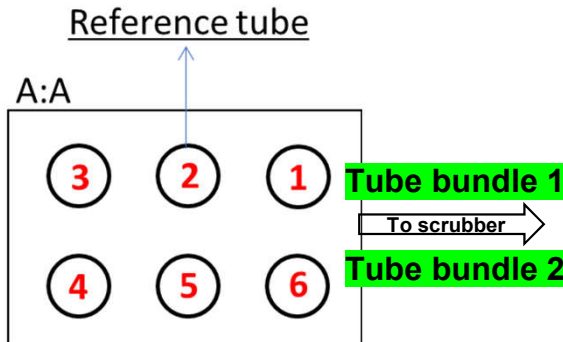
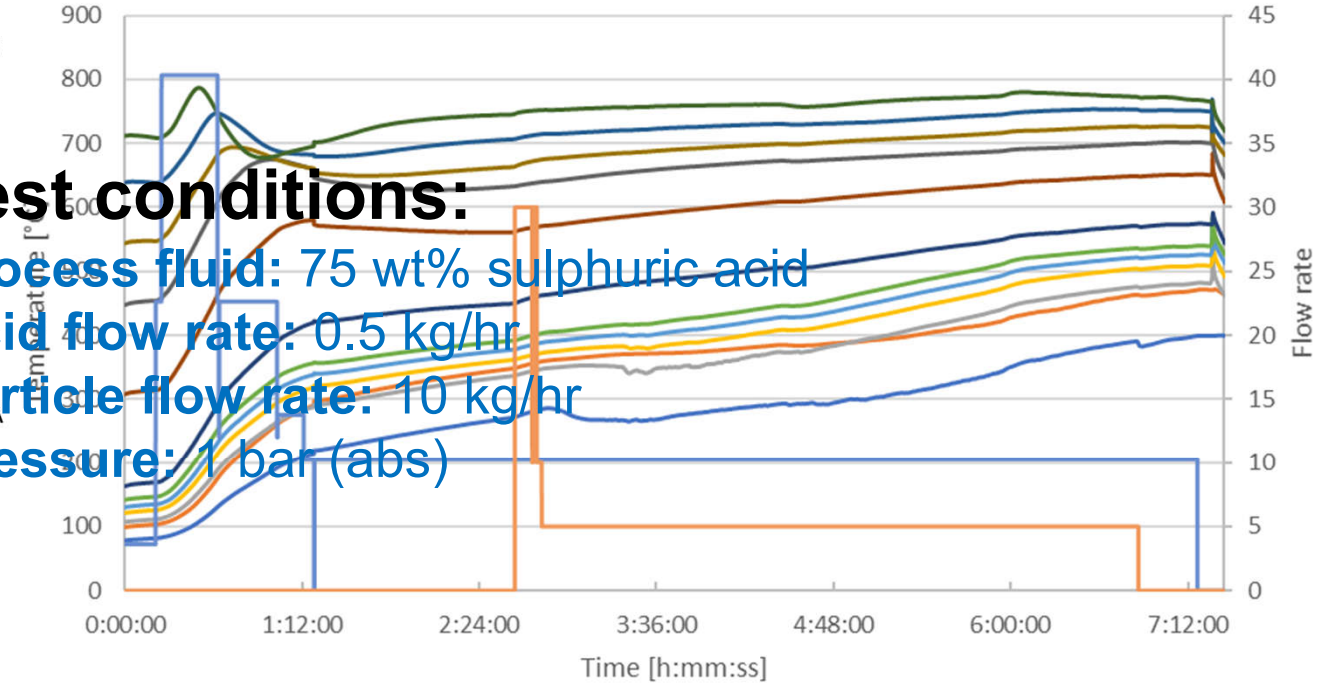
# Particles-heated decomposition reactor

## Temperature distribution – Chemical test (02.06.2021)



### Test conditions:

- Process fluid: 75 wt% sulphuric acid
- Acid flow rate: 0.5 kg/hr
- Particle flow rate: 10 kg/hr
- Pressure: 1 bar (abs)



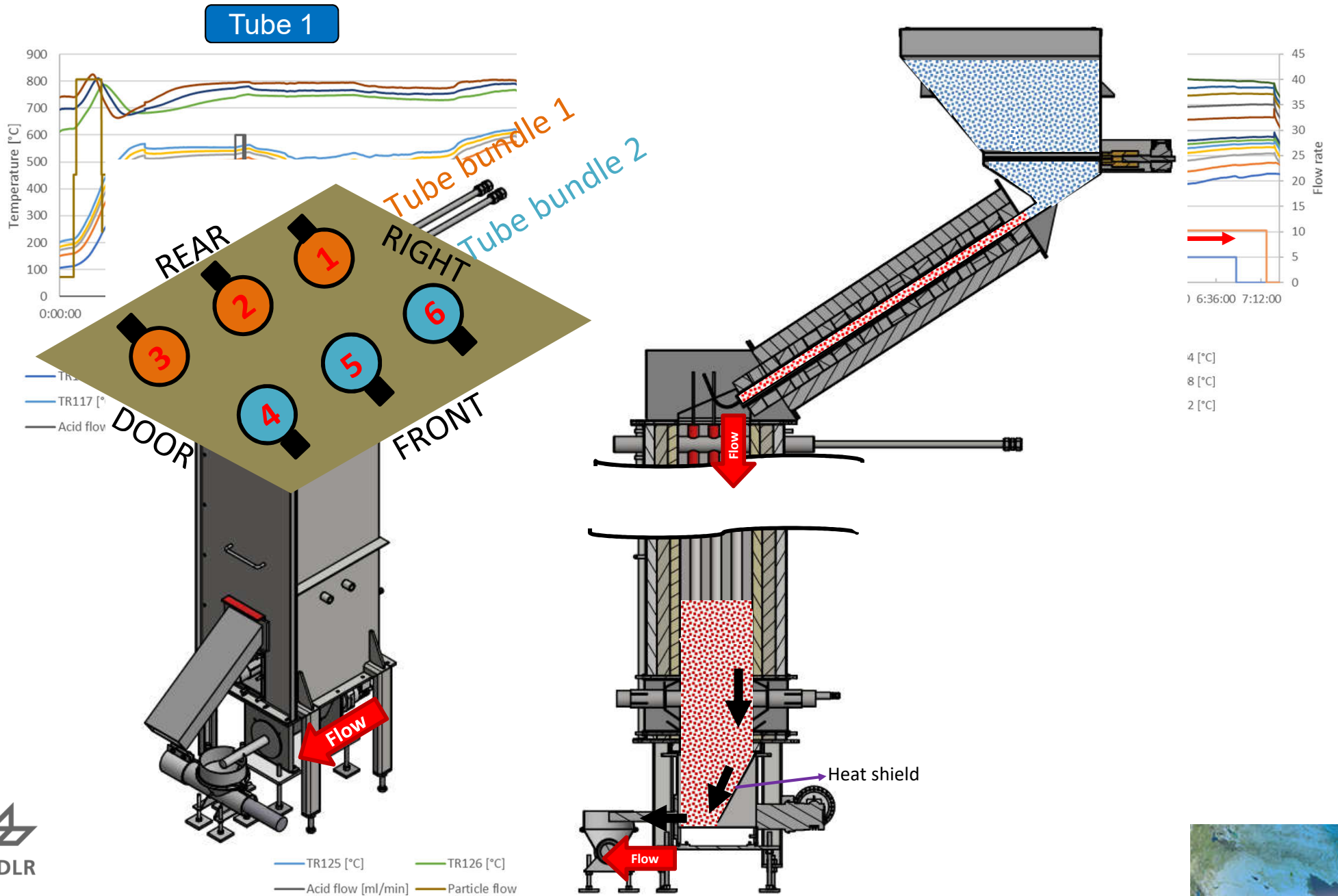
- Position 0
- Position 1
- Position 2
- Position 3
- Position 4
- Position 5
- Position 6
- Position 7
- Position 8
- Position 9
- Position 10
- Position 11
- Particle flow [kg/h]
- Acid flow [ml/min]

Position i: Average [Position i(tube1) ..... Position i(tube 6)]



# Particles-heated decomposition reactor

## Temperature distribution – Chemical test (02.06.2021)

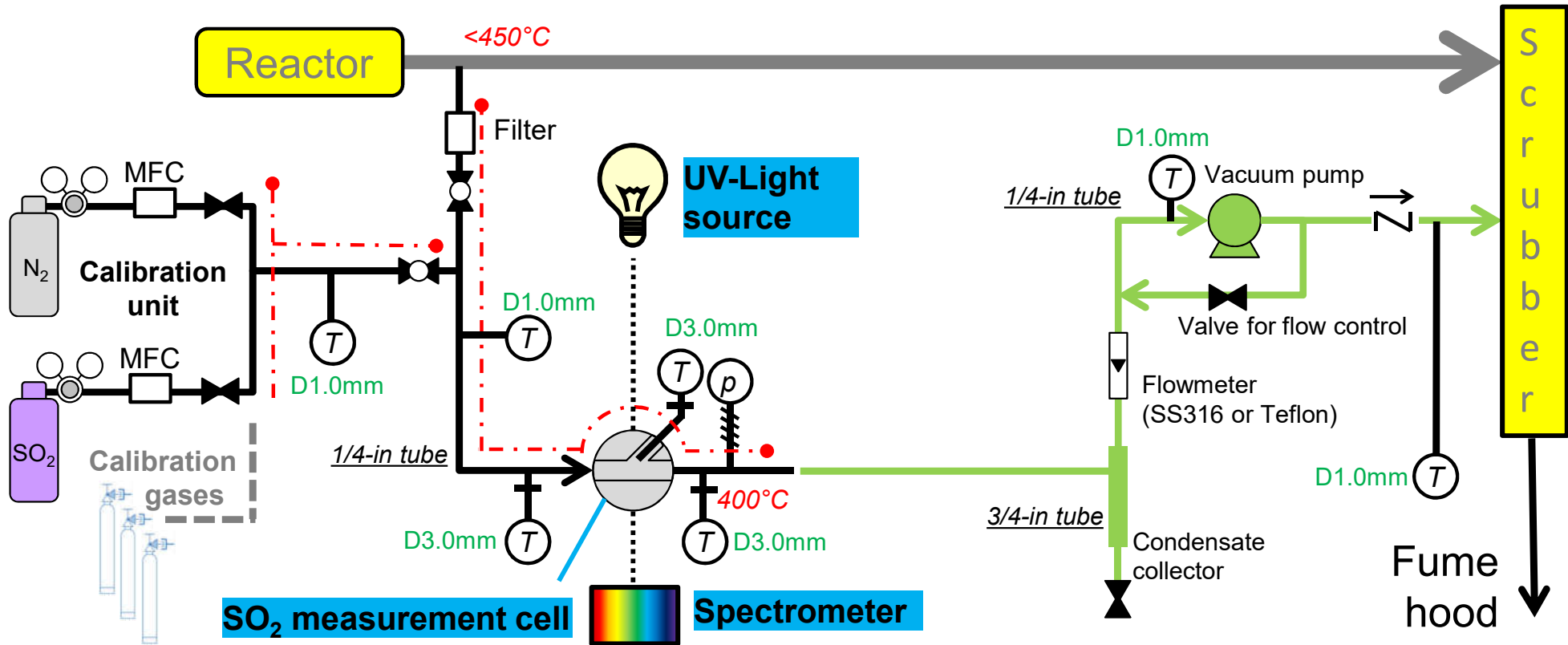




# Particles-heated decomposition reactor

## Development of a SO<sub>2</sub> measurement system

Schematics of the SO<sub>2</sub> measurement system

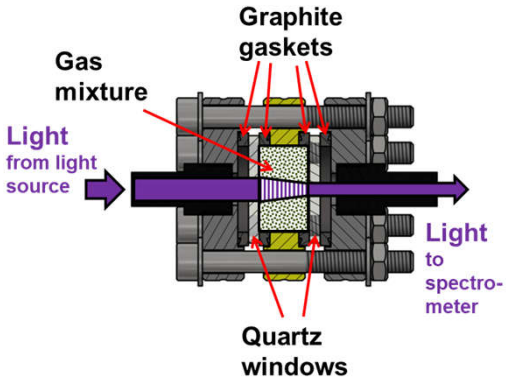
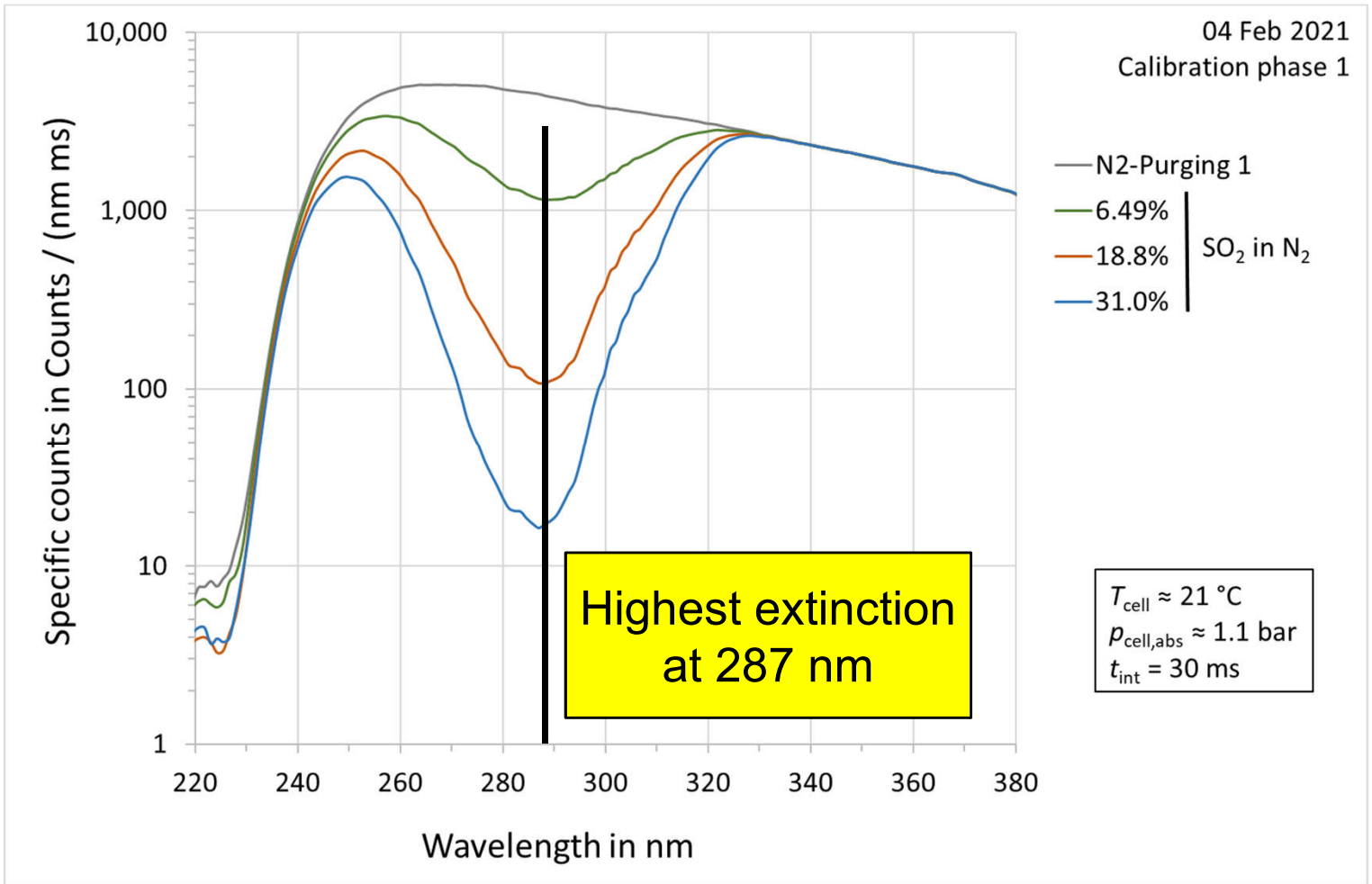


- 1.4404(SS316)
- Teflon(PFA)
- ..... Fibre optics
- - - - • Heating band
- ⊗ Solenoid valve
- ⊗ normal valve
- T temperature measurement (thermocouple type K)
- p pressure measurement
- MFC mass flow controller



# Particles-heated decomposition reactor

## Calibration of SO<sub>2</sub> measurement system at room temperature



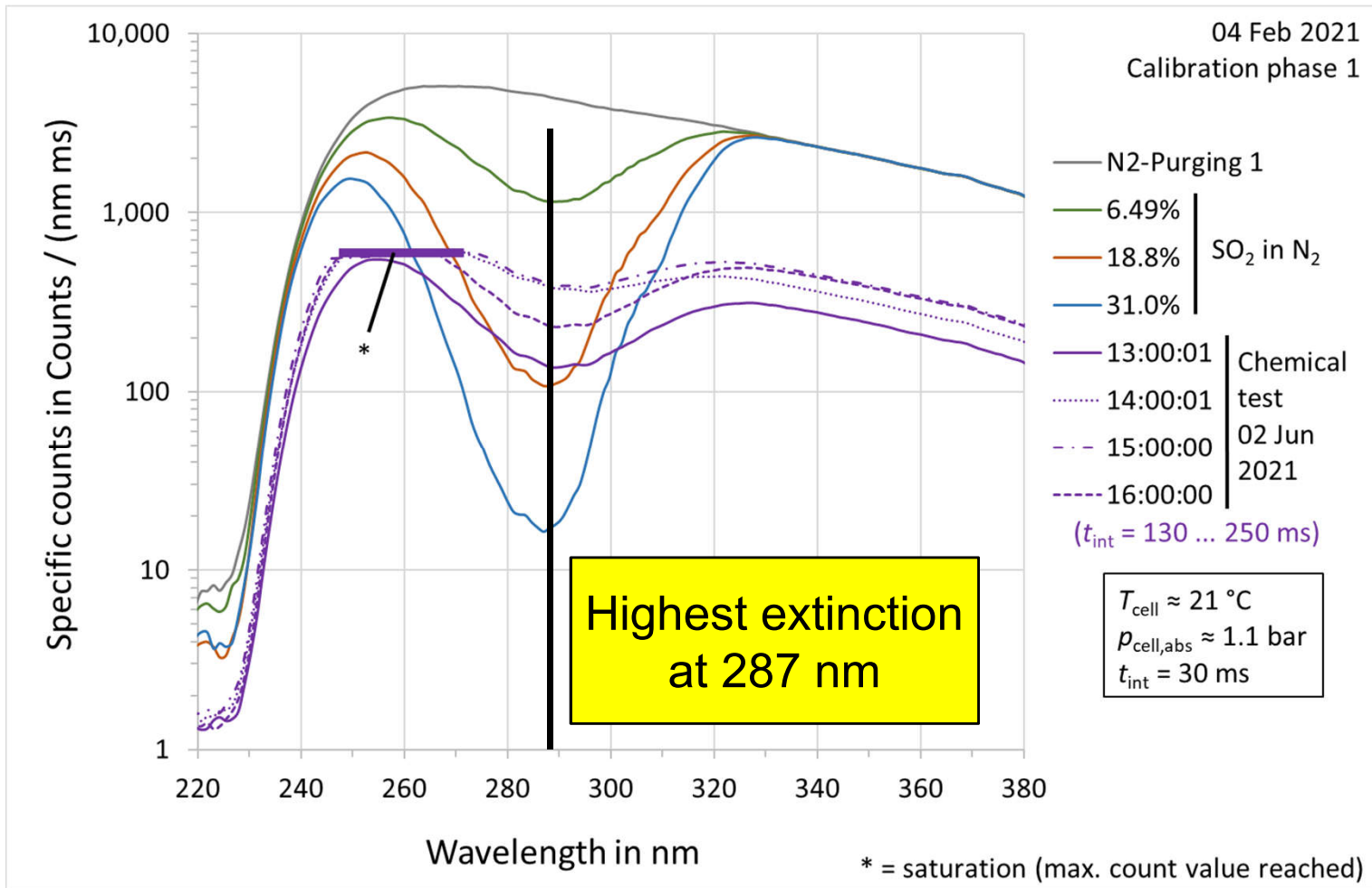
Plausible decrease of counts with molar SO<sub>2</sub> fraction.

Very stable spectra.



# Particles-heated decomposition reactor

## Comparison with spectra gained during chemical test



Spectra gained during chemical test feature shape characteristic for SO<sub>2</sub>.

**SO<sub>2</sub> was produced!**

Overheating of measurement cell led to detachment of optical parts:



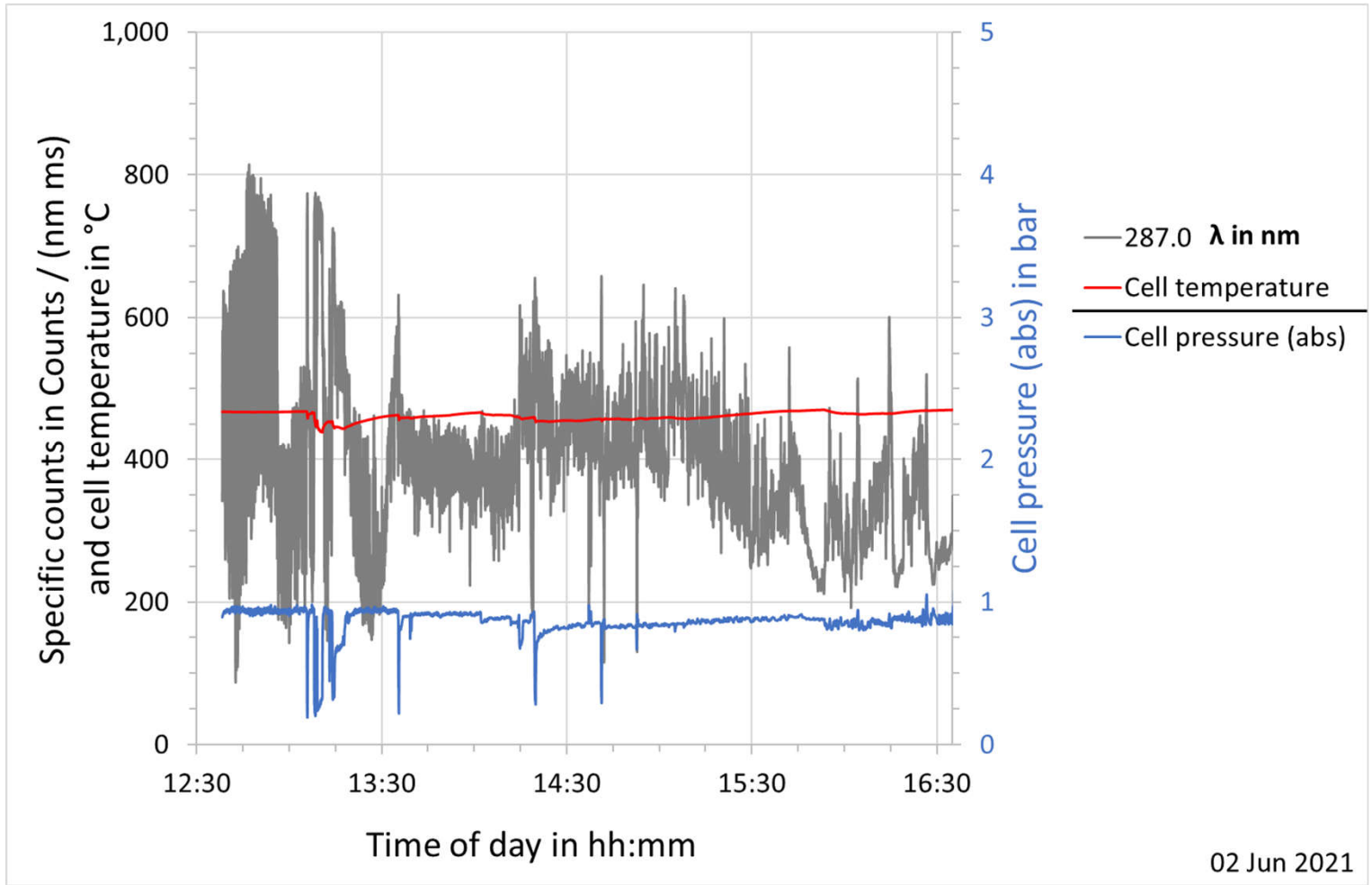
Reference spectrum not valid, quantification planned in further tests.





# Particles-heated decomposition reactor

## Evolution of specific counts during chemical test



SO<sub>2</sub> produced the whole test day.

Fairly stable production level of SO<sub>2</sub>.



# Particles-heated decomposition reactor

## Conclusions

- A new first of its kind particle based sulphuric acid decomposition reactor was developed
- A dedicated particle heater to heat up the particle bed up to 900°C was designed, developed and validation-tested, delivering particles of temperature higher than 870°C to the reactor
- A new optical SO<sub>2</sub> measurement system was developed for gas analysis at 400°C
- Thermal test was performed to check the thermal performance of the reactor (70% by the end of the test)
- A chemical test was conducted demonstrating sulphuric acid evaporation and sulphur trioxide splitting
  - Temperatures in the zone of the former were much above the boiling point securing complete evaporation
  - Temperatures in catalyst zone need to be increased by improving the insulation. However, minimum temperature required for Fe<sub>2</sub>O<sub>3</sub> at 75w% acid is 720°C to avoid sulphate formation [1], which was reached
- Further chemical tests will still be performed after reworking the reactor



# Thank you for your attention

