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Lothar Holitzner :: Designing Engineer :: Paul Scherrer Institut

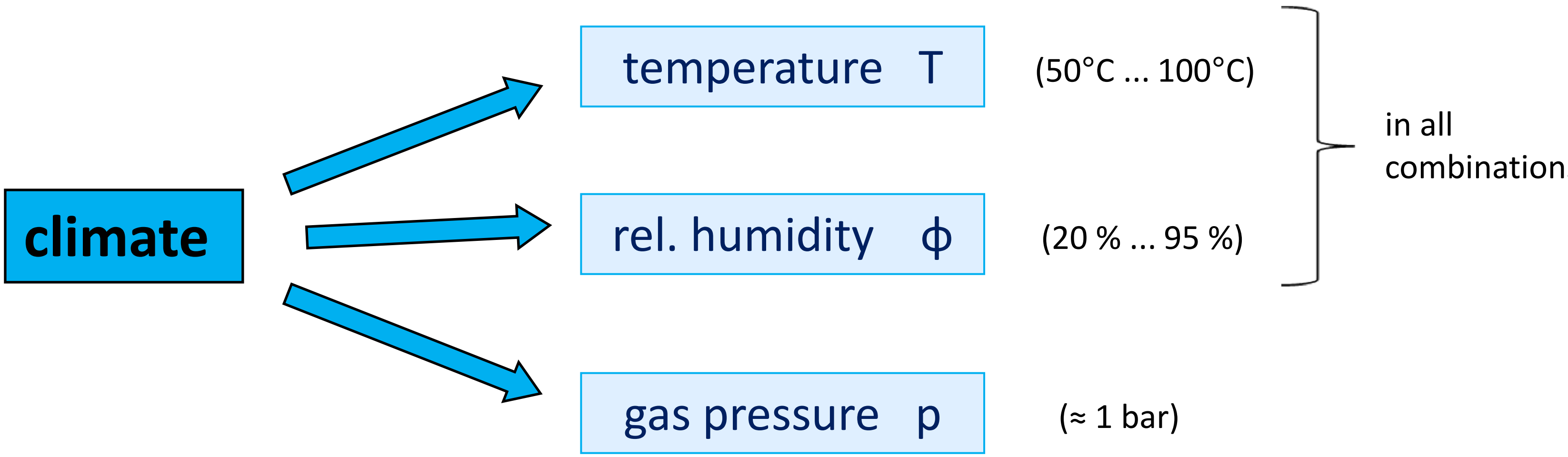
Advanced Sample Environment Design - An Example for SINQ instrument SANS-II

A Climate Chamber with Fast Humidity and Temperature Response

7th Design and Engineering of Neutron Instruments Meeting 2018

Technical specification: Climate elements

Climate inside the chamber



required precision:

$\Delta T = \pm 1°C$
$\Delta \phi = \pm 2\ \%$

Our Application

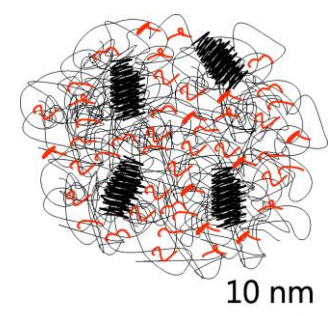
Small-angle Neutron Scattering (SANS)

The instrument :

Neutron guide hall

SINQ

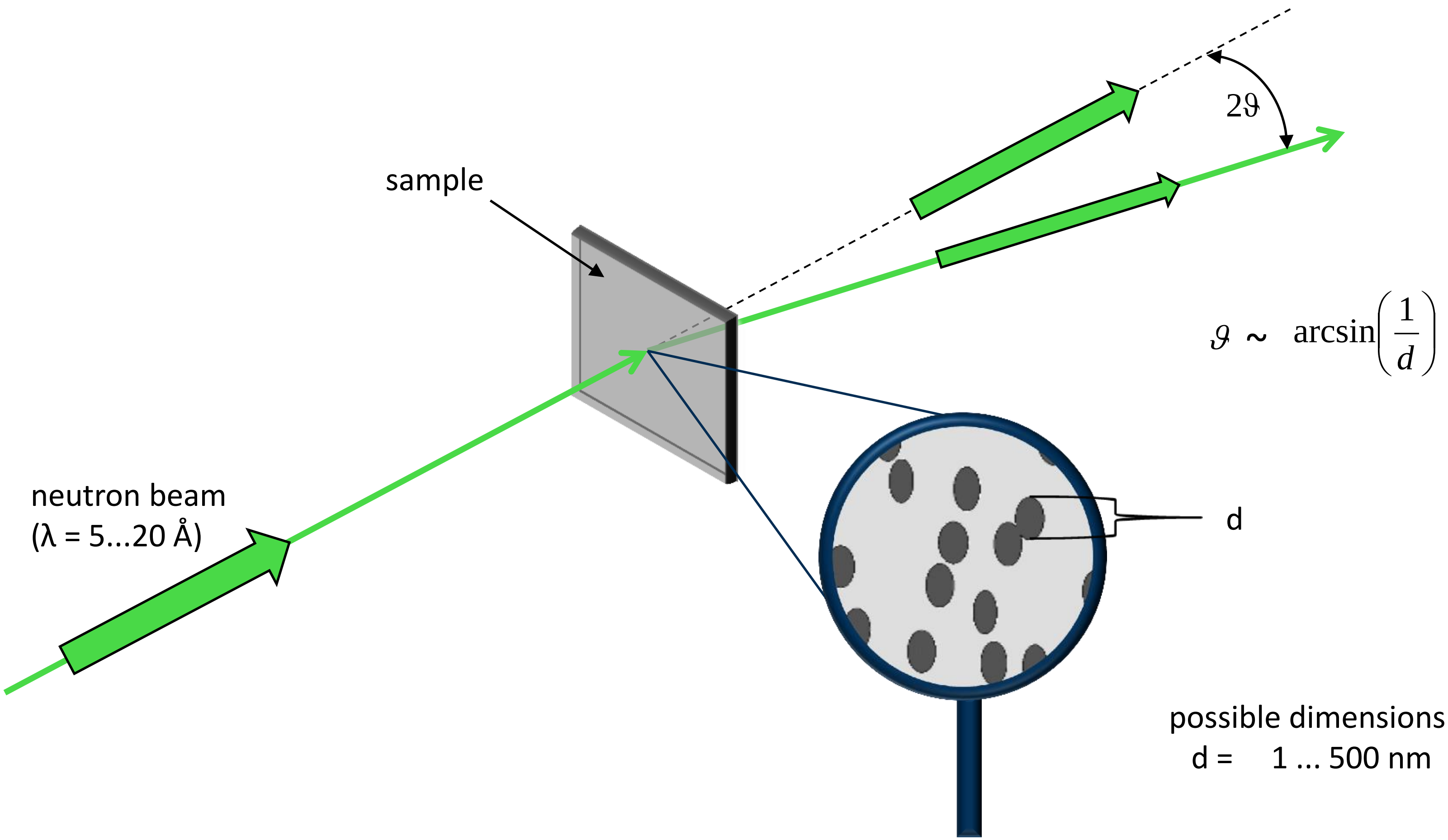
Sample example



Our Application

Small-angle Neutron Scattering (SANS)

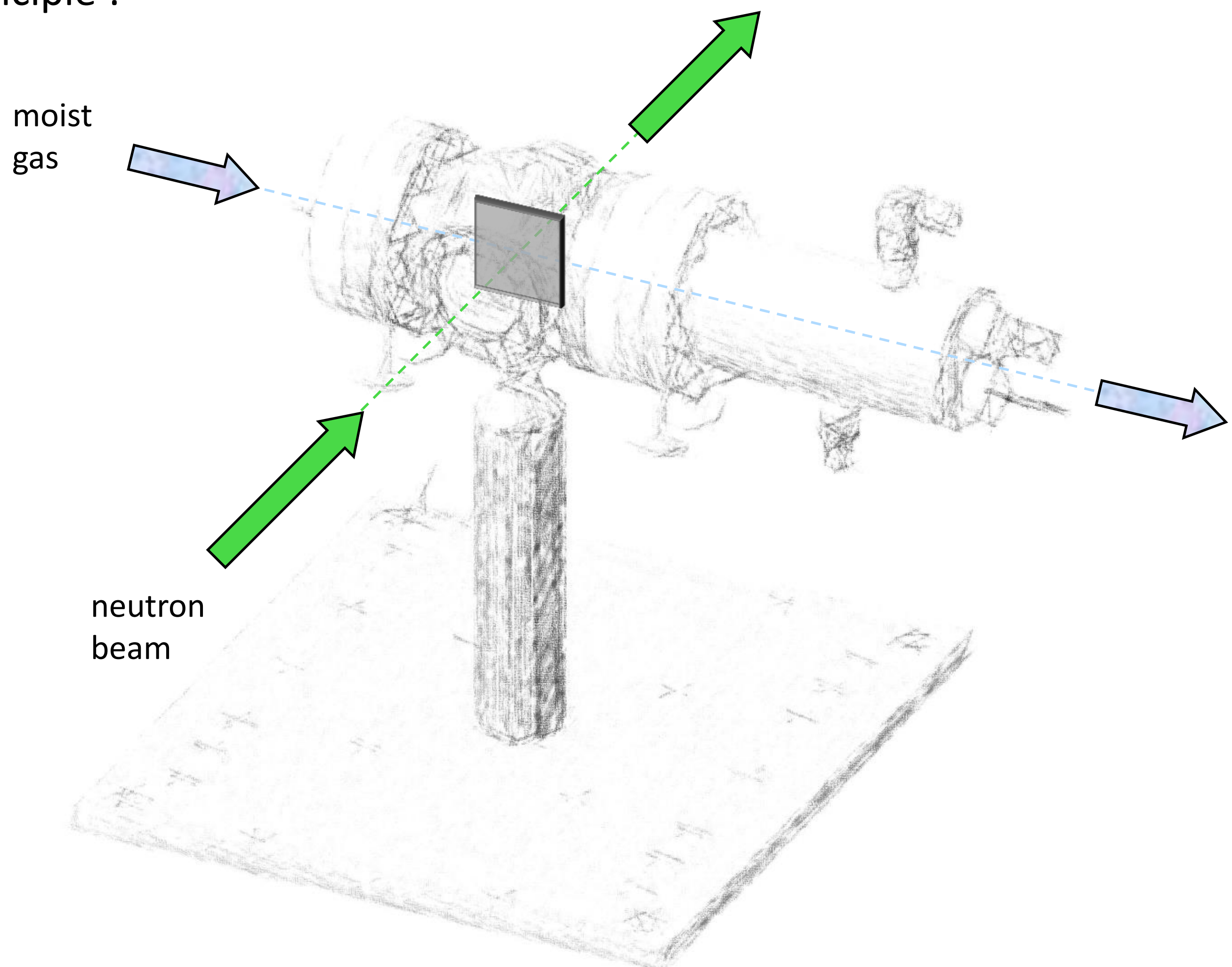
The principle :



Experimental setup

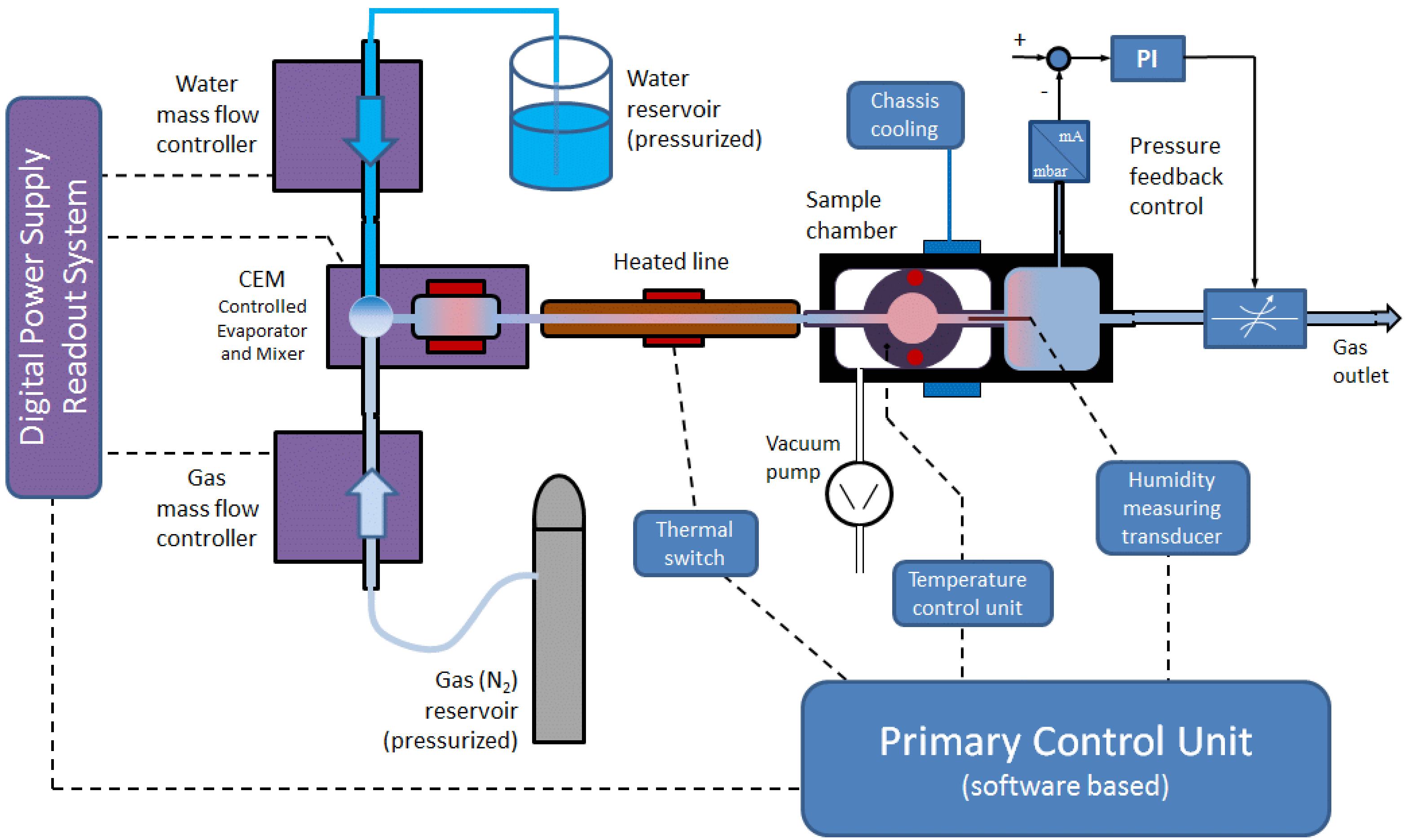
Experimental setup with device sketch

The principle :



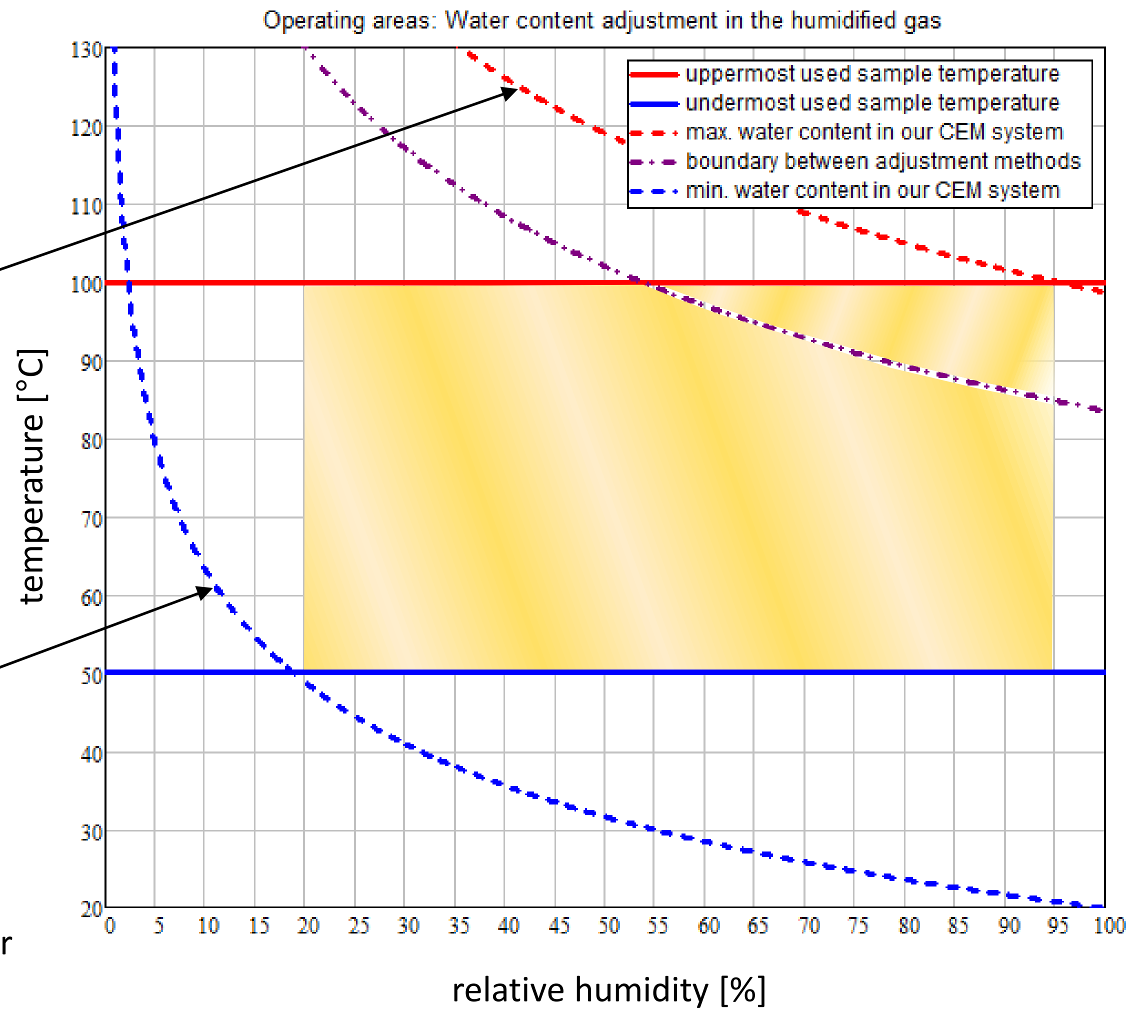
System overview

Climate chamber system



Operation overview

Operating area and limits , Curves with constant water content



max.
water content
$X_{CEM.max}$ = 6.000

min.
water content
$X_{CEM.min}$ = 0.014

$p_{process}$ = 1070 mbar

$$\frac{X_{CEM.\max}}{X_{CEM.\min}} = 428$$

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Operation overview

Water content (= moisture content x_{vap})

Ideal gas law

$$p_{w.vap} \cdot V = m_{w.vap} \frac{R_{uni}}{M_{water}} \cdot T$$

$$p_{dry.gas} \cdot V = m_{dry.gas} \frac{R_{uni}}{M_{dry.gas}} \cdot T$$

water content (mixing ratio)

$$X = \frac{m_{water}}{m_{dry.gas}}$$

Relative humidity

$$\phi = \frac{p_{w.vap}}{p_{w.vap.sat}}$$

$$X(p,T,\phi) = \frac{M_{water}}{M_{dry.gas}} \cdot \frac{p_{w.vap.sat}(T)}{\frac{p}{\phi} - p_{w.vap.sat}(T)}$$

(main equation)

Partial pressure sum

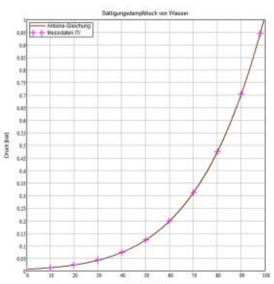
$$p = \sum p_i = p_{w.vap} + p_{dry.gas}$$

p – pressure [bar]

T – temperature [°C]

ϕ – rel. humidity (RH)

M – molar mass



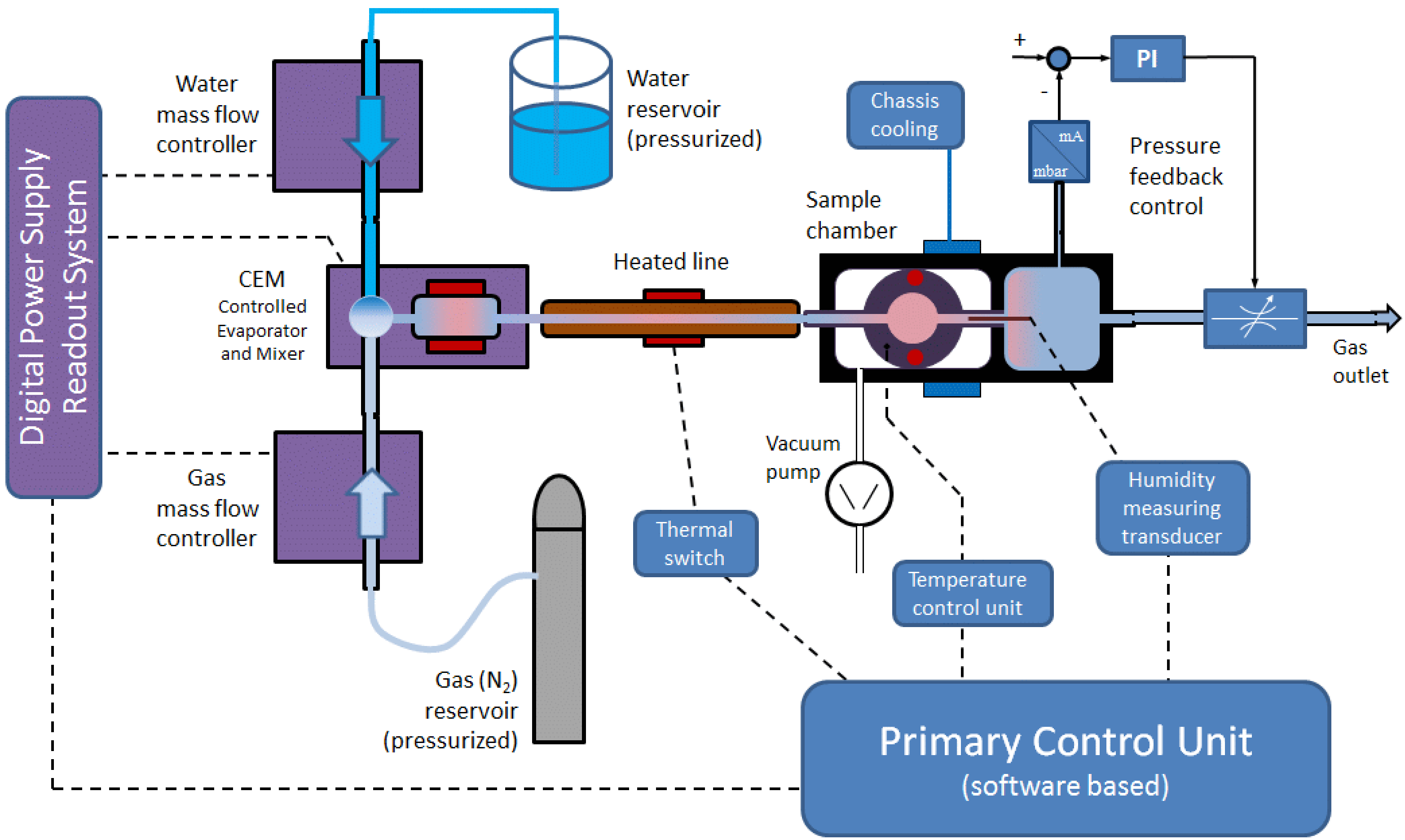
Vapor pressure of water

$$\log\left(\frac{p_{w.vap.sat}}{bar}\right) = 5.19625 - \frac{1730.630}{233.426 + \frac{T}{°C}}$$

(Antoine equation for 1 -100°C)

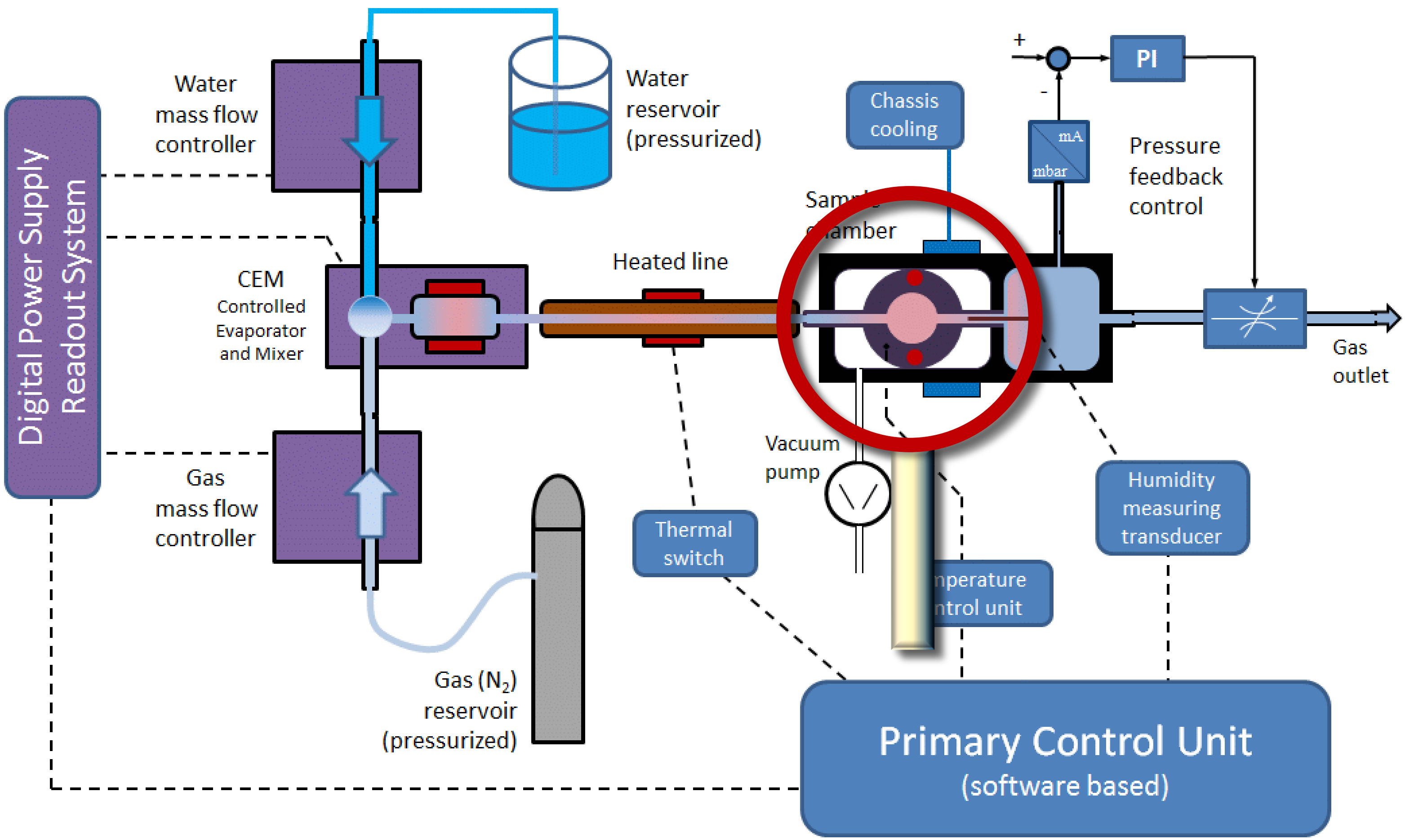
System overview

Climate chamber system



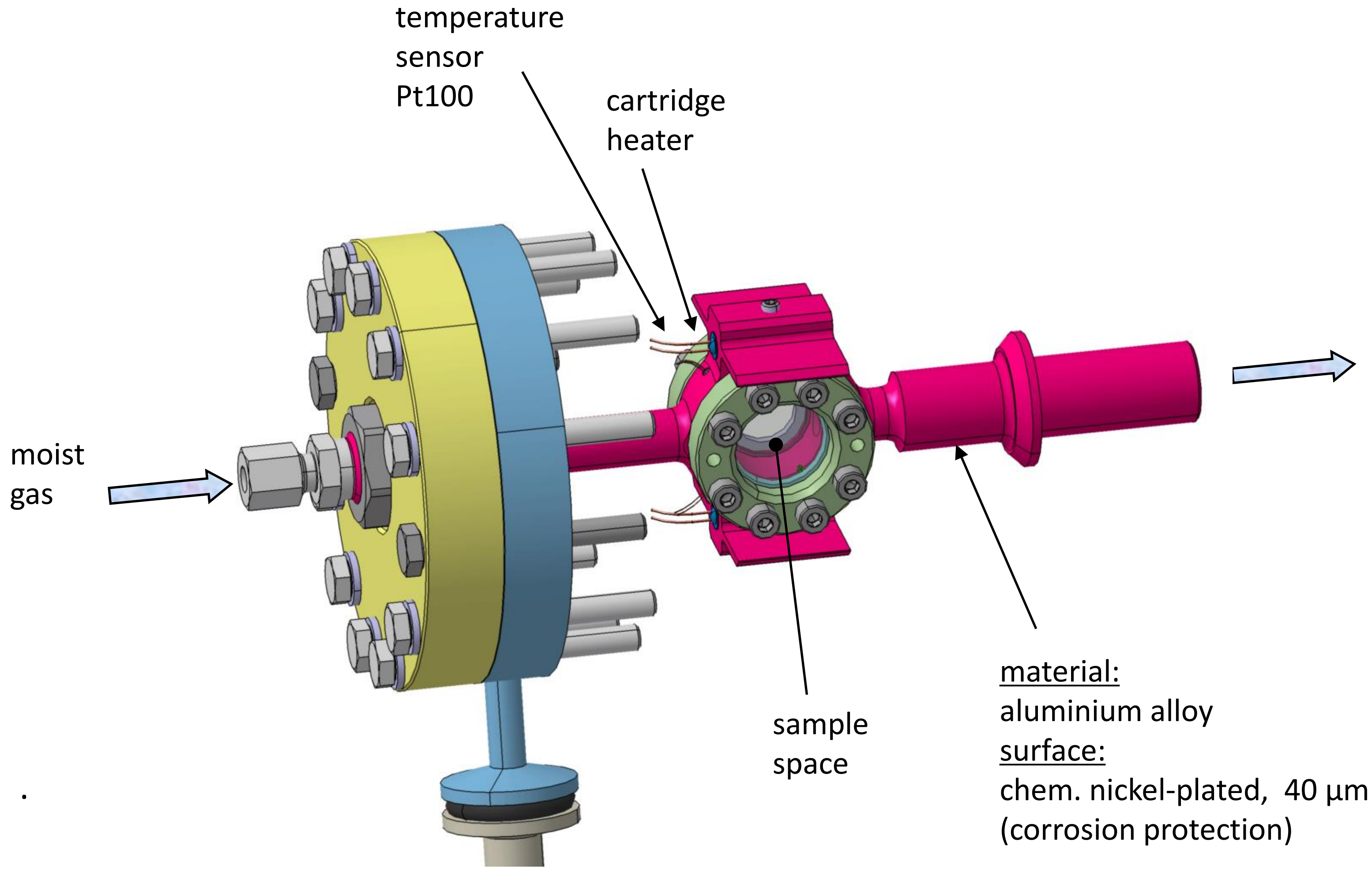
System overview

Climate chamber system



Device details

Internal chamber



<u>material:</u>
aluminium alloy
<u>surface:</u>
chem. nickel-plated, 40 µm
(corrosion protection)

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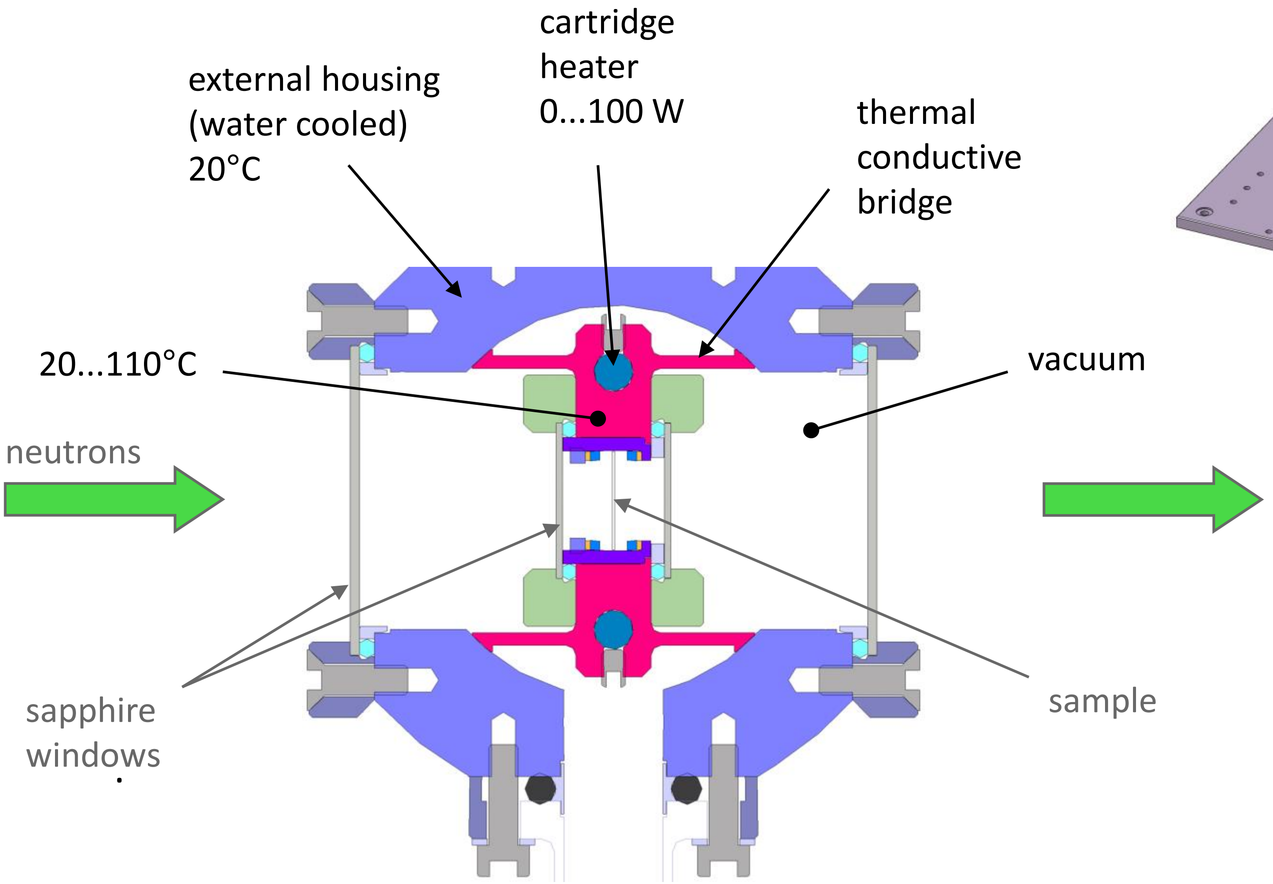
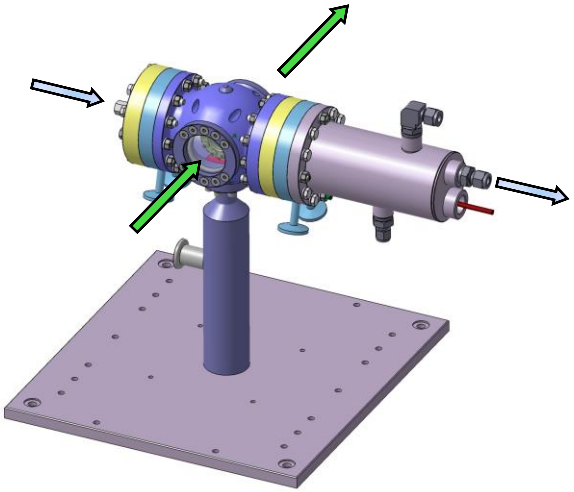
Device details

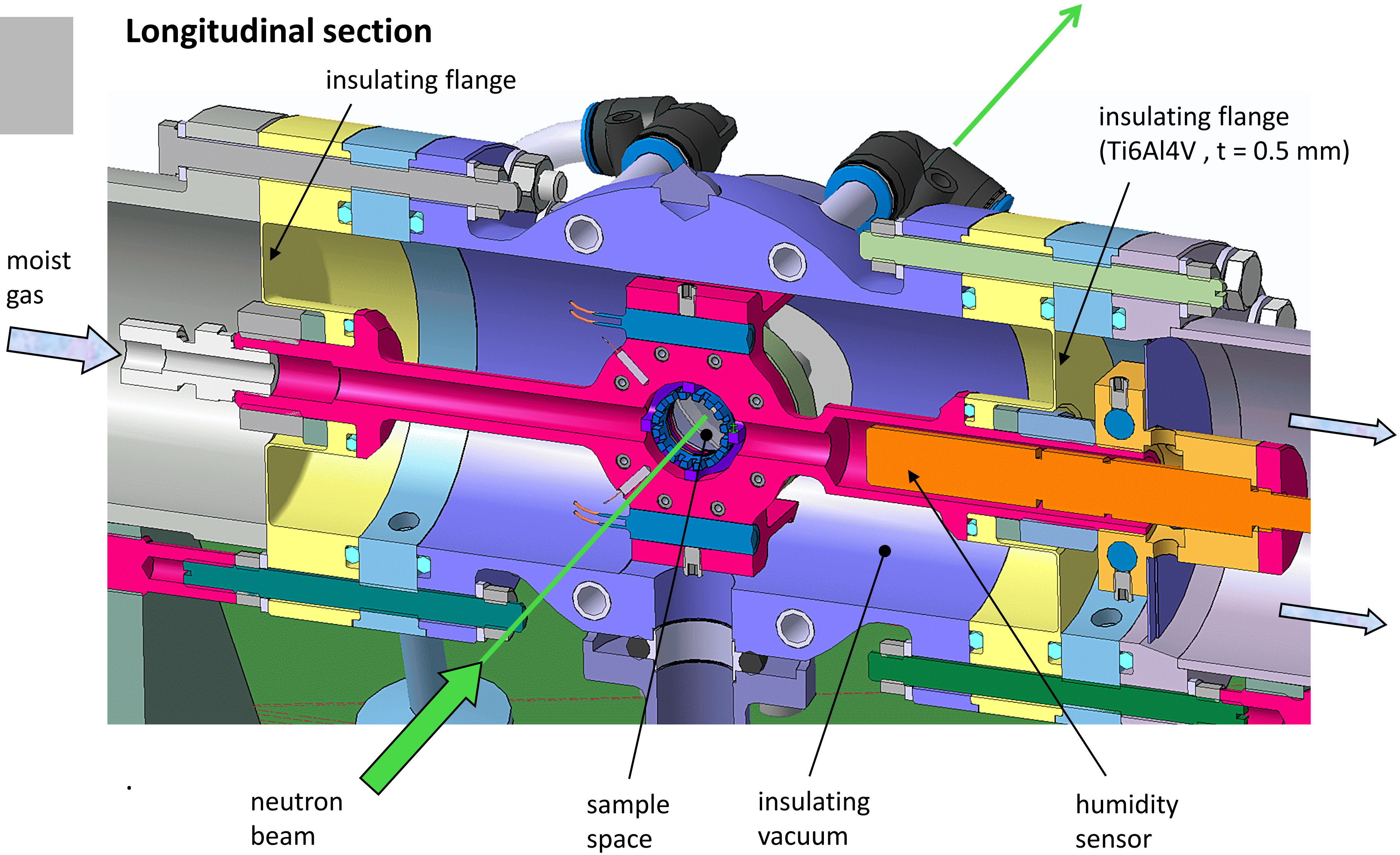
Fast heating & cooling the internal chamber





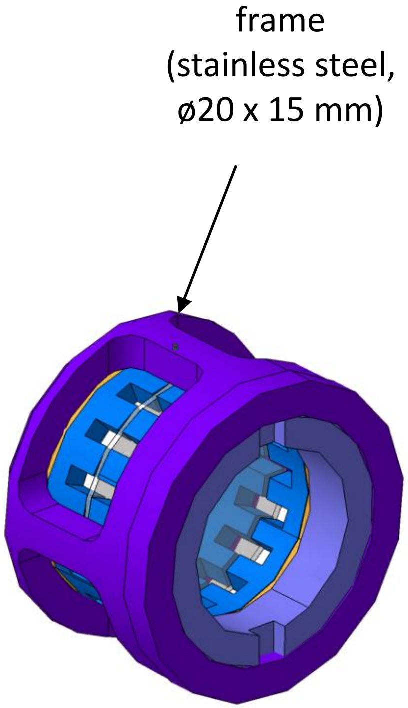
Device details

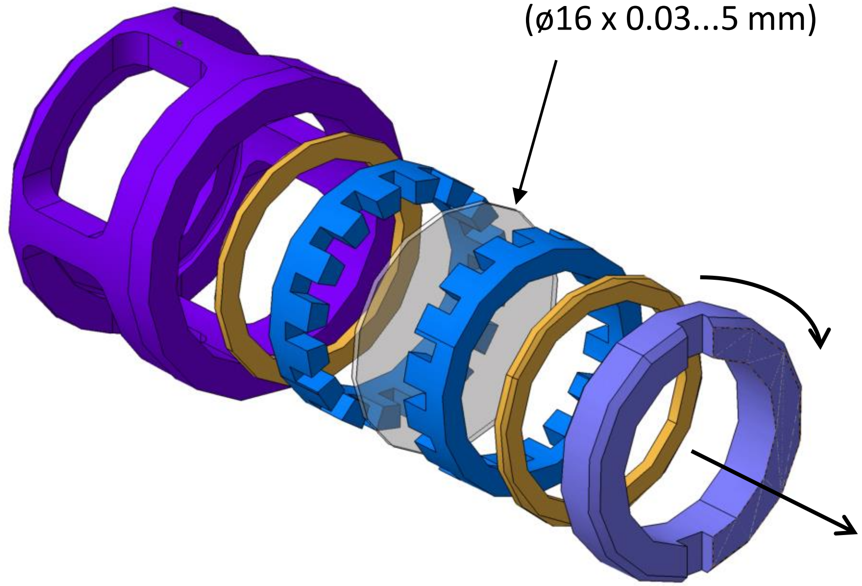
Longitudinal section



Device details

Sample holder





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Design criteria

Our goals

1. Climate precision: temperature $\Delta T = \pm 1°C$
   rel. humidity $\Delta\phi = \pm 2\ \%$

2. Gas flow distribution nearby sample surface: as uniform as possible

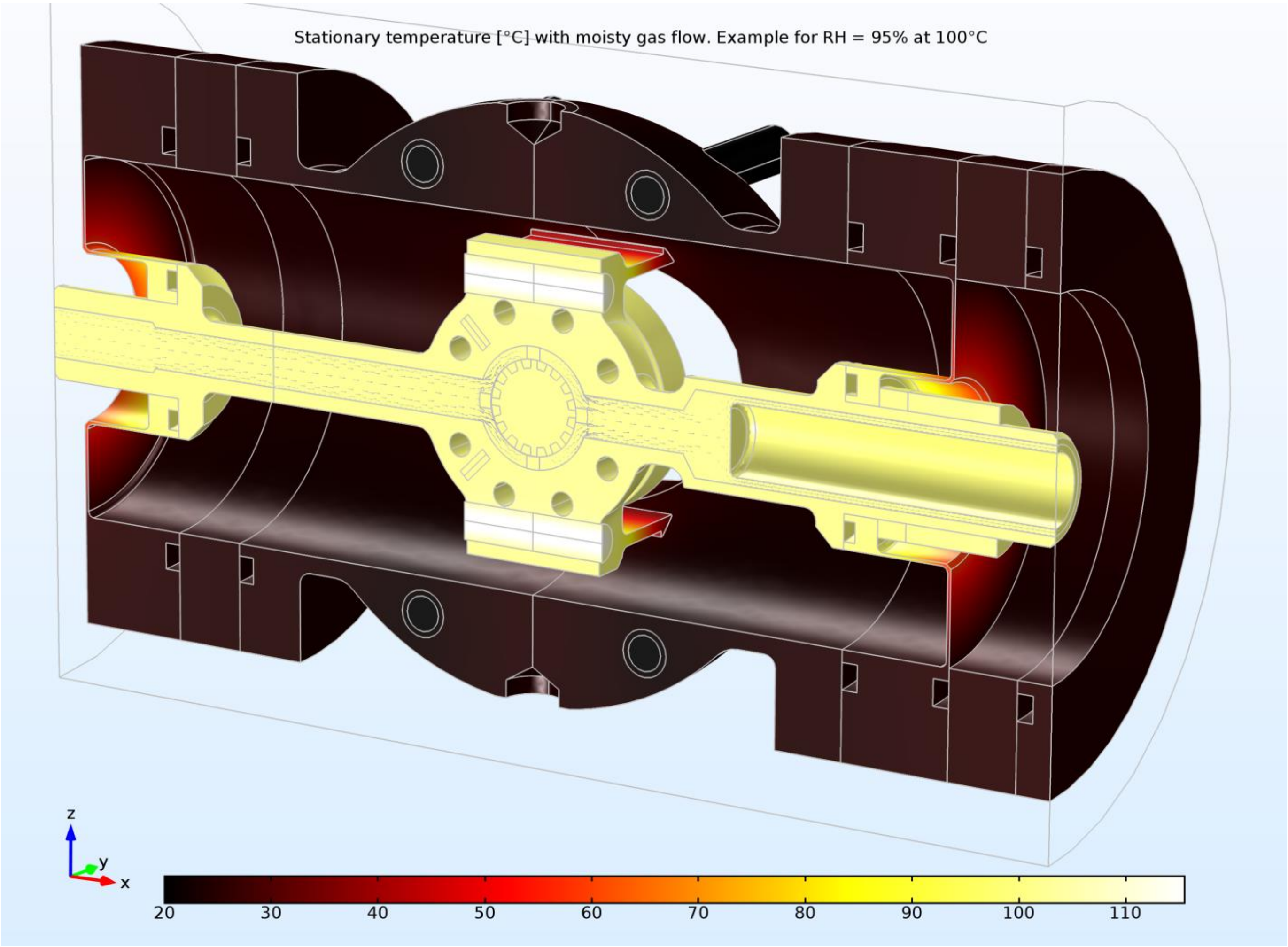
3. Change of temperature: similar time constants for heating and cooling
   → improved temperature controllability

4. Change of climate: as fast as possible

Stationary temperature distribution

❶ Climate precision

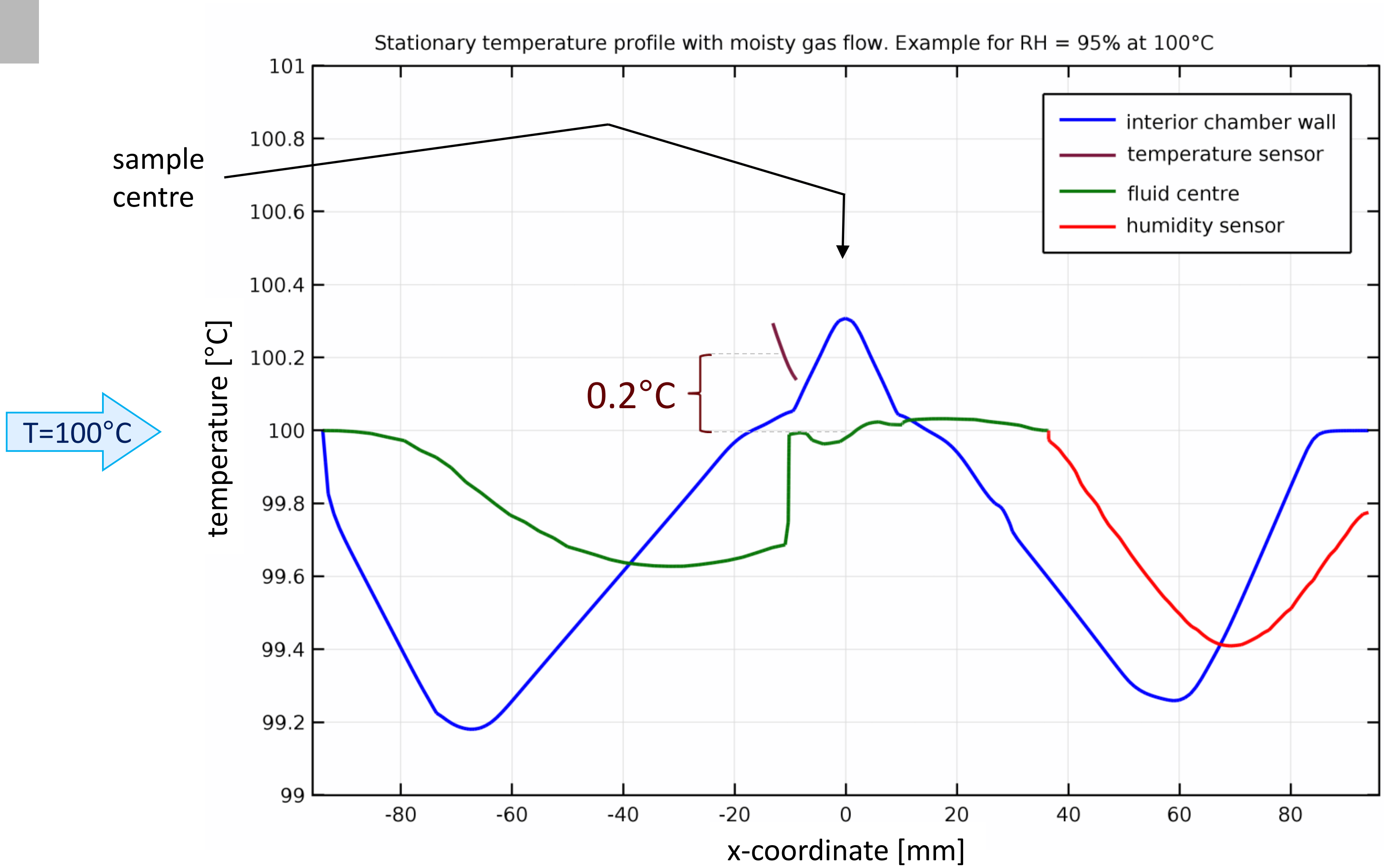
Example with moisty gas flow for ϕ = 95% at 100°C



Stationary temperature distribution

1 Climate precision

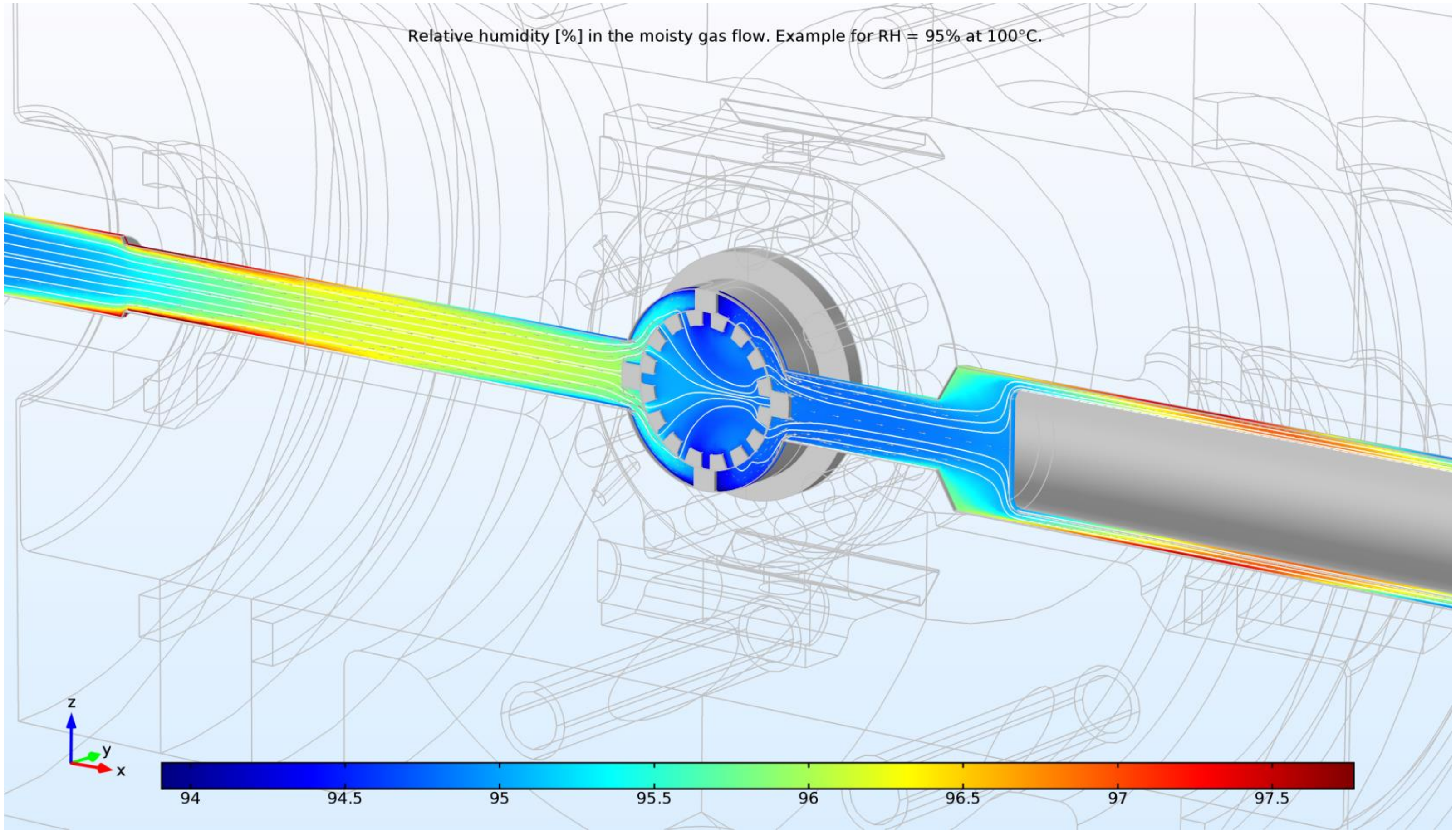
Temperature scan in gas flow direction (x-axis) , climate: ϕ = 95% at 100°C



Humidity distribution

❶ Climate precision

Example with moist gas flow for ϕ = 95% at 100°C



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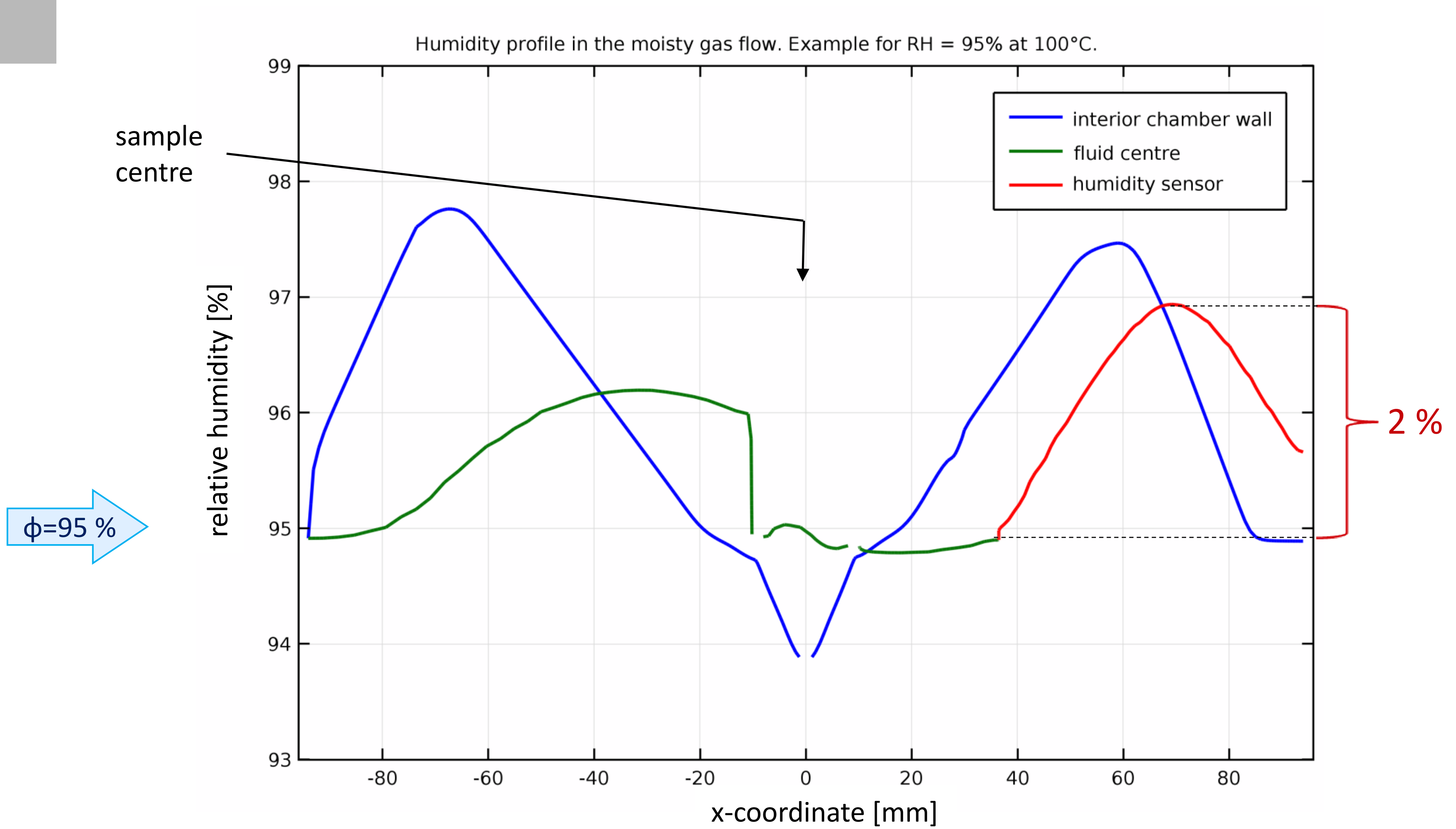
Humidity distribution

❶ Climate precision

Humidity scan in gas flow direction (x-axis) , climate: ϕ = 95% at 100°C

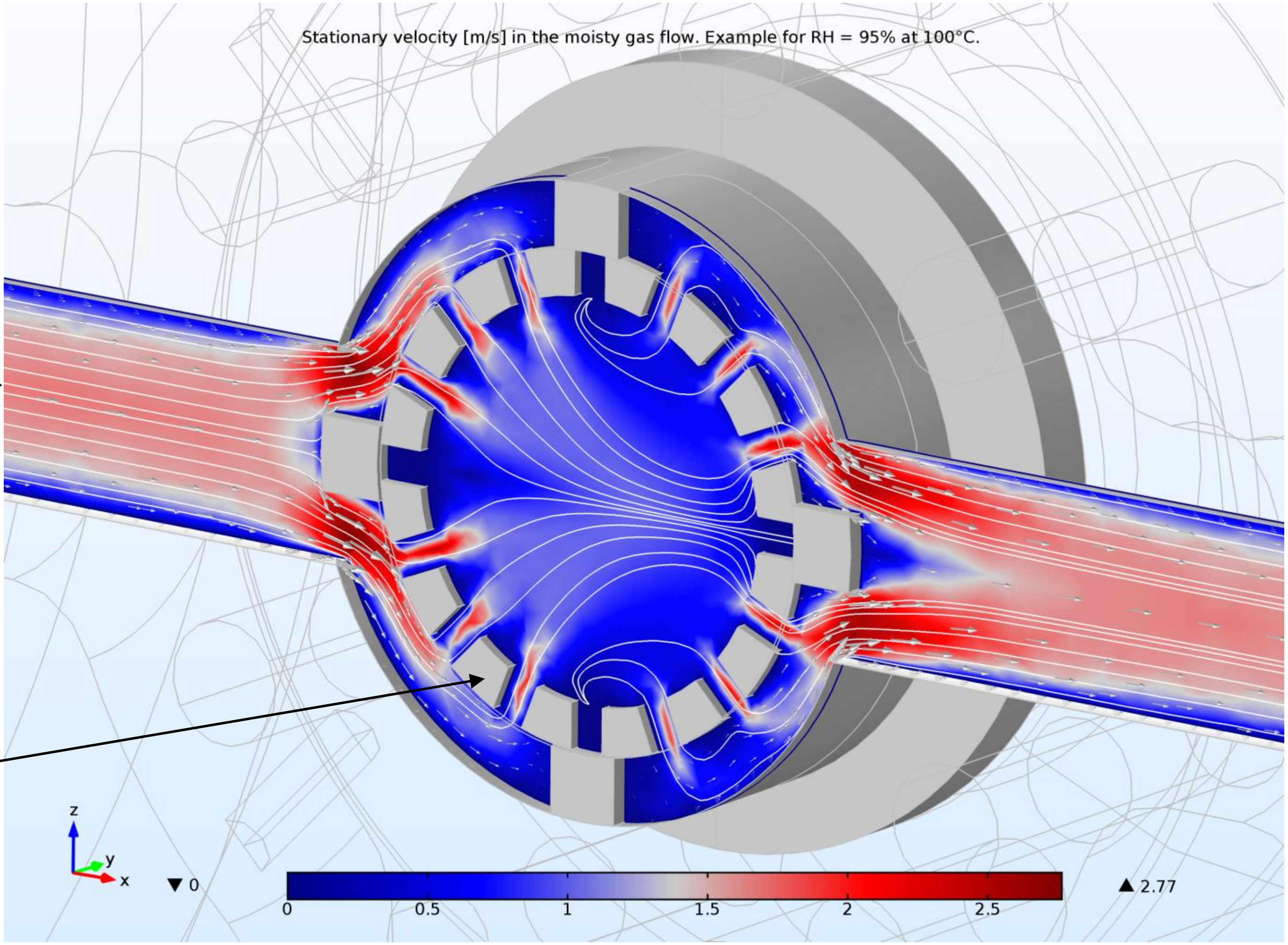


Stationary velocity distribution

❷ Gas flow distribution nearby sample surface

Example with moisty gas flow for ϕ = 95% at 100°C

moist gas

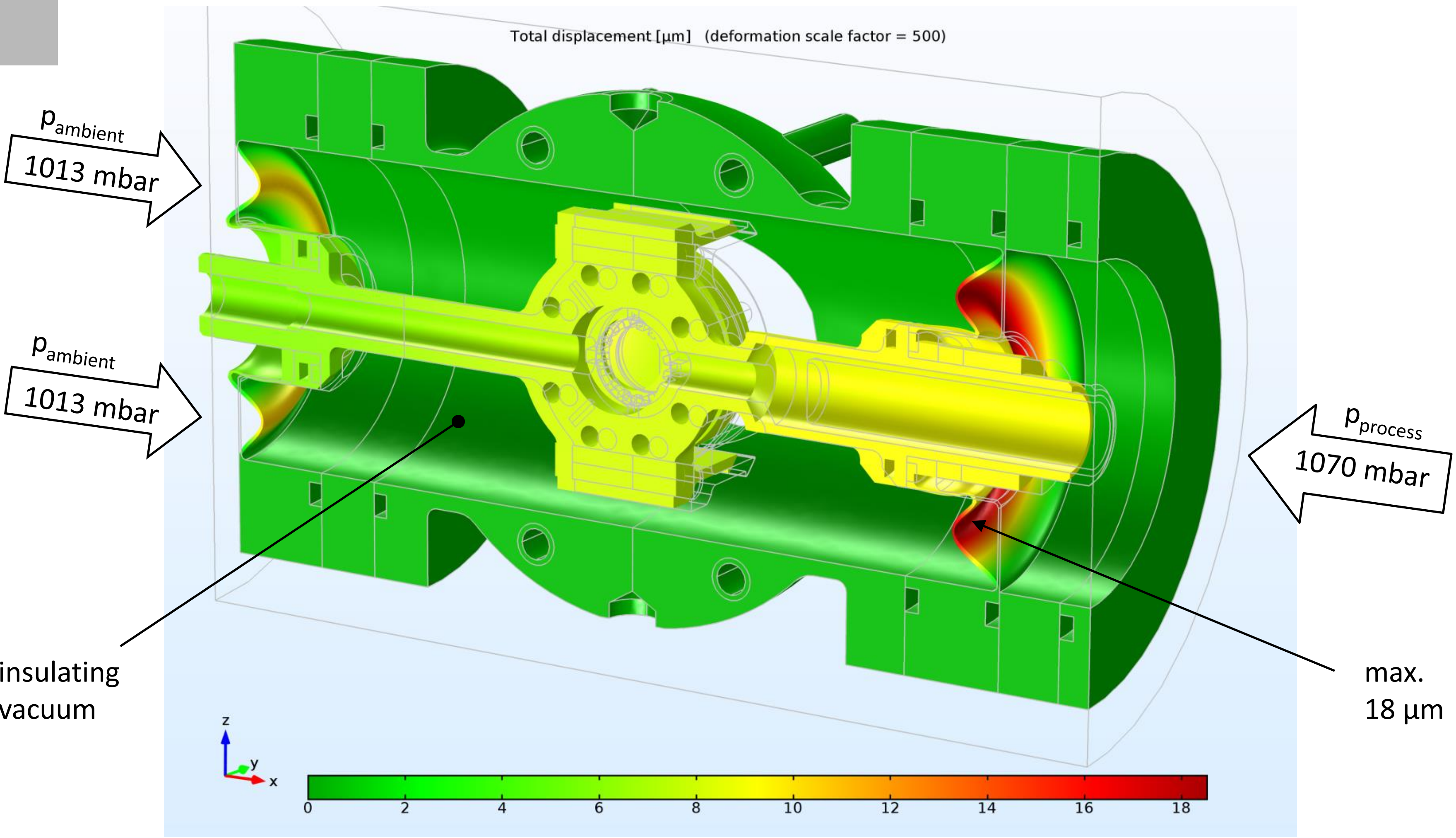


sample holder

Total displacement

❸ Change of temperature

Chamber with insulating vacuum, under process pressure, at 100°C



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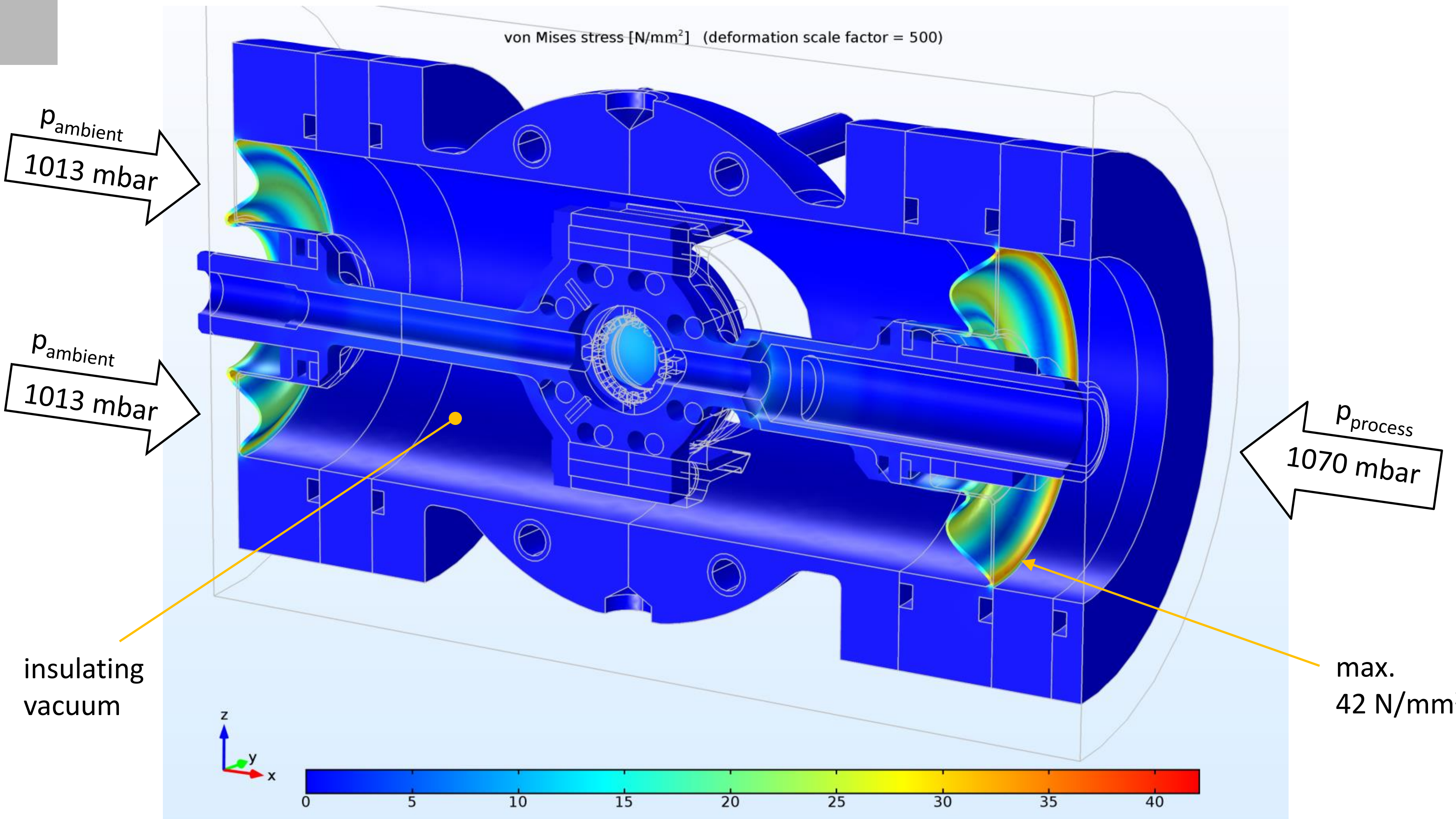
Mechanical stress

❸ Change of temperature

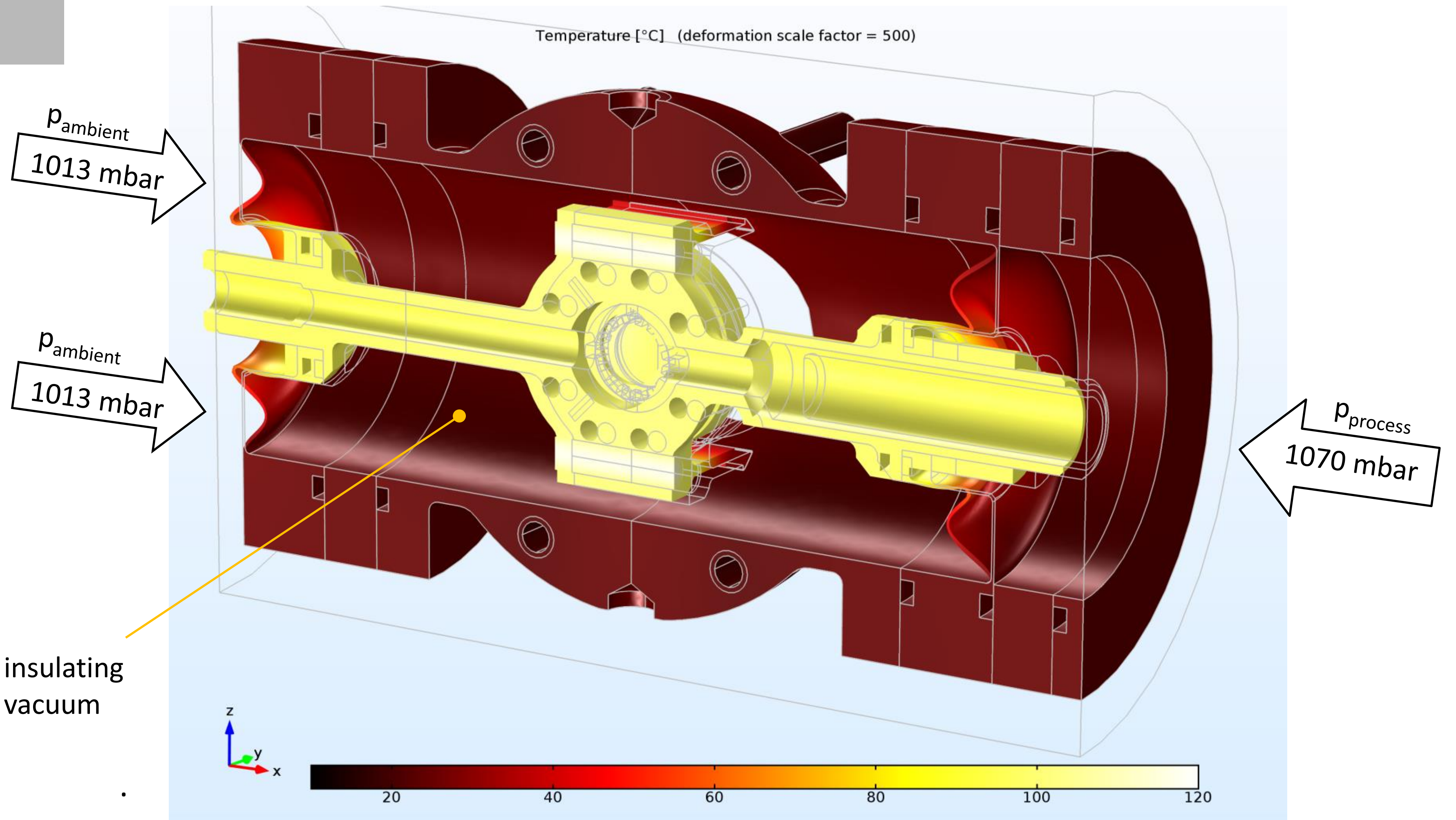
Chamber with insulating vacuum, under process pressure, at 100°C



Temperature distribution and displacement

❸ Change of temperature

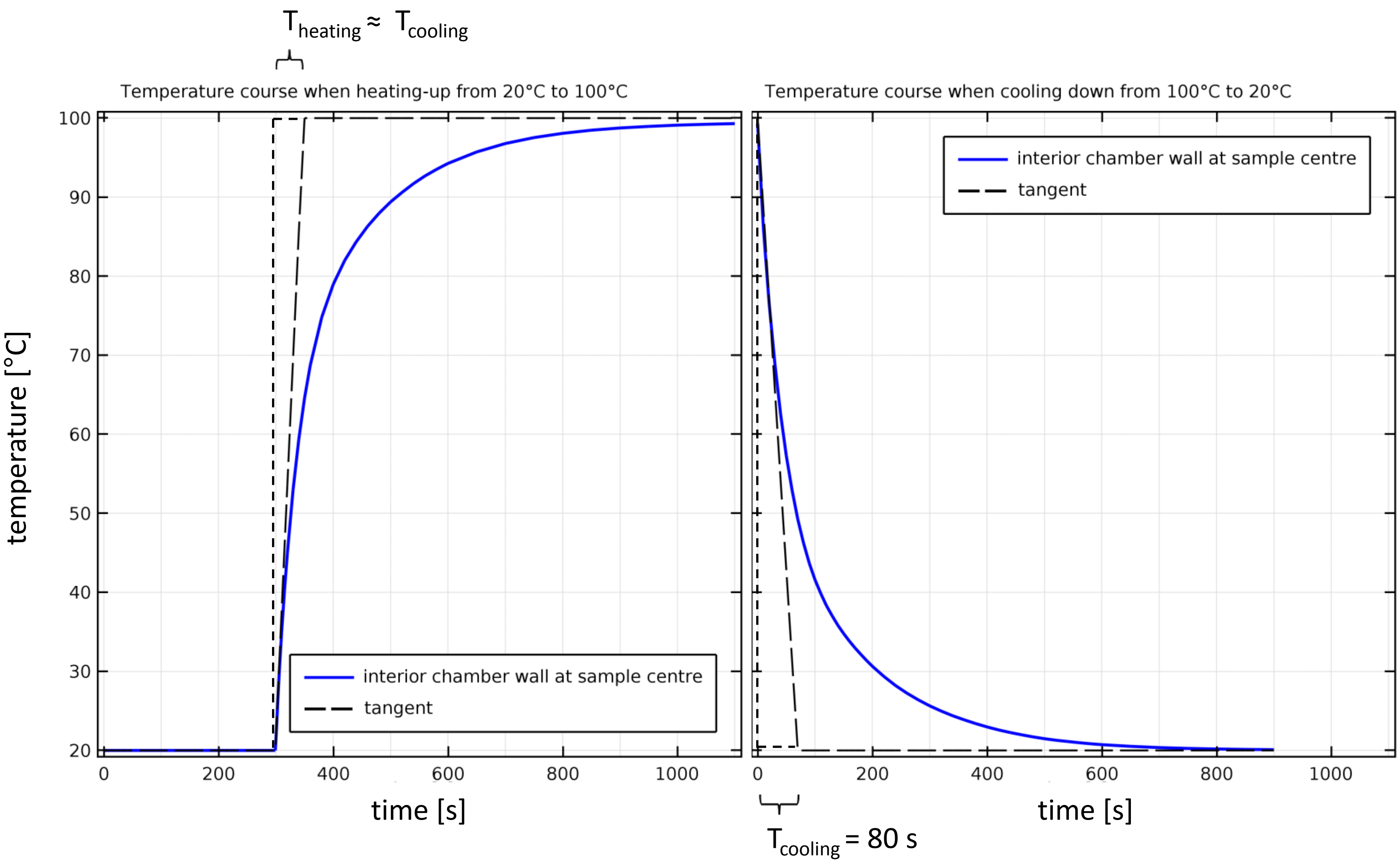
Chamber with insulating vacuum, under process pressure, at 100°C



Temperature change

❸ Change of temperature

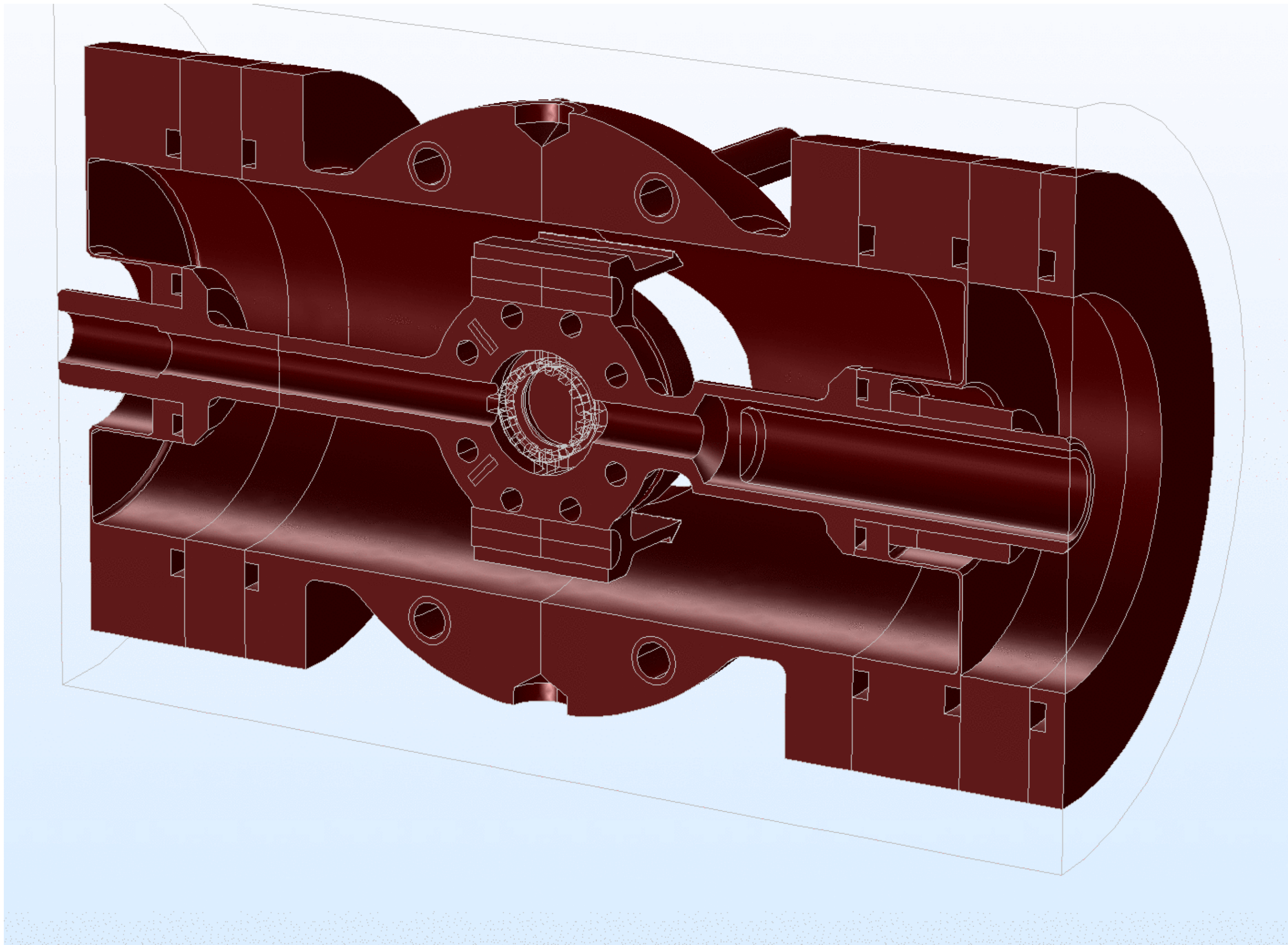
Step function response when heating-up or cooling-down



Temperature distribution and displacement

❸ Change of temperature

Starting sequence (insulating vacuum → process pressure → temperature change)

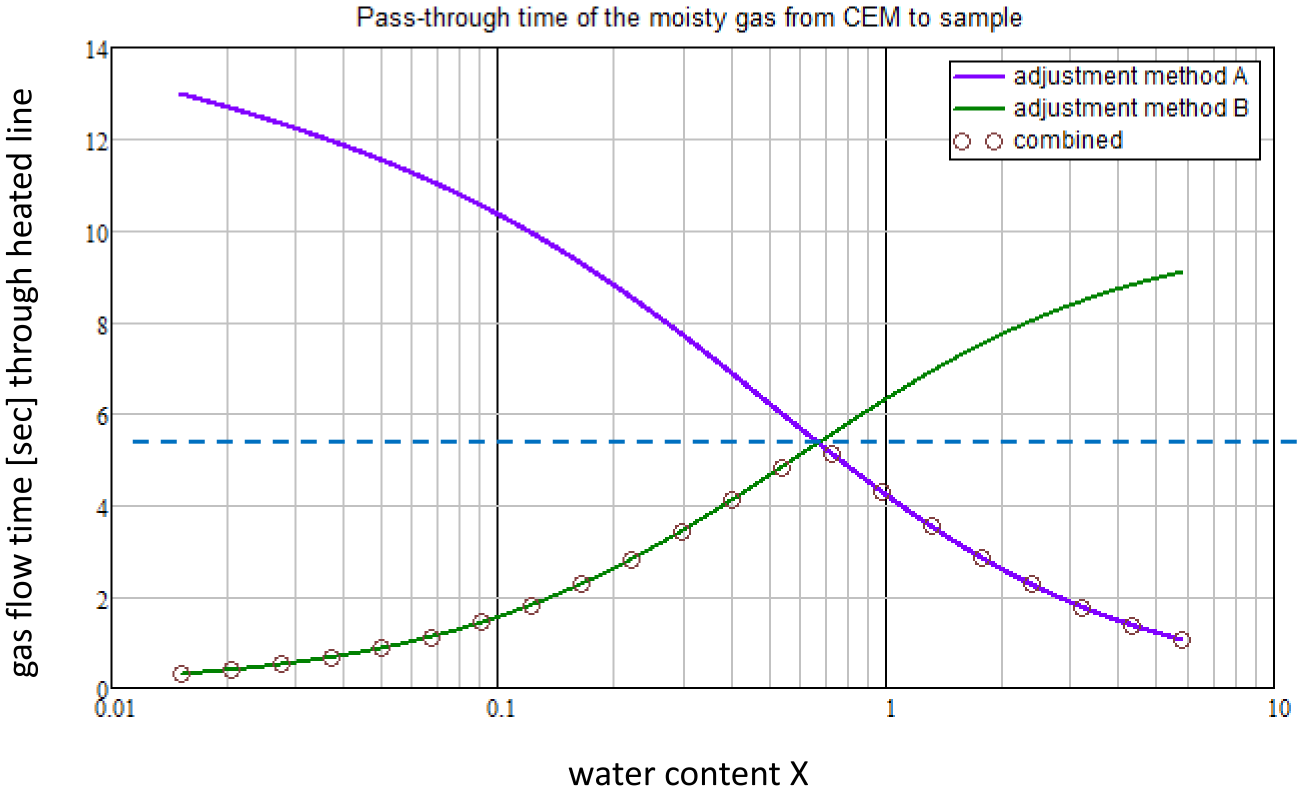


1. switch on vacuum pump
2. apply process pressure
3. set sample temperature

Humidity change

❹ Change of climate

Moisty gas flow time from CEM to sample → humidity response



5,4 sec (transfer line)

\+ **8 sec** (sensor)

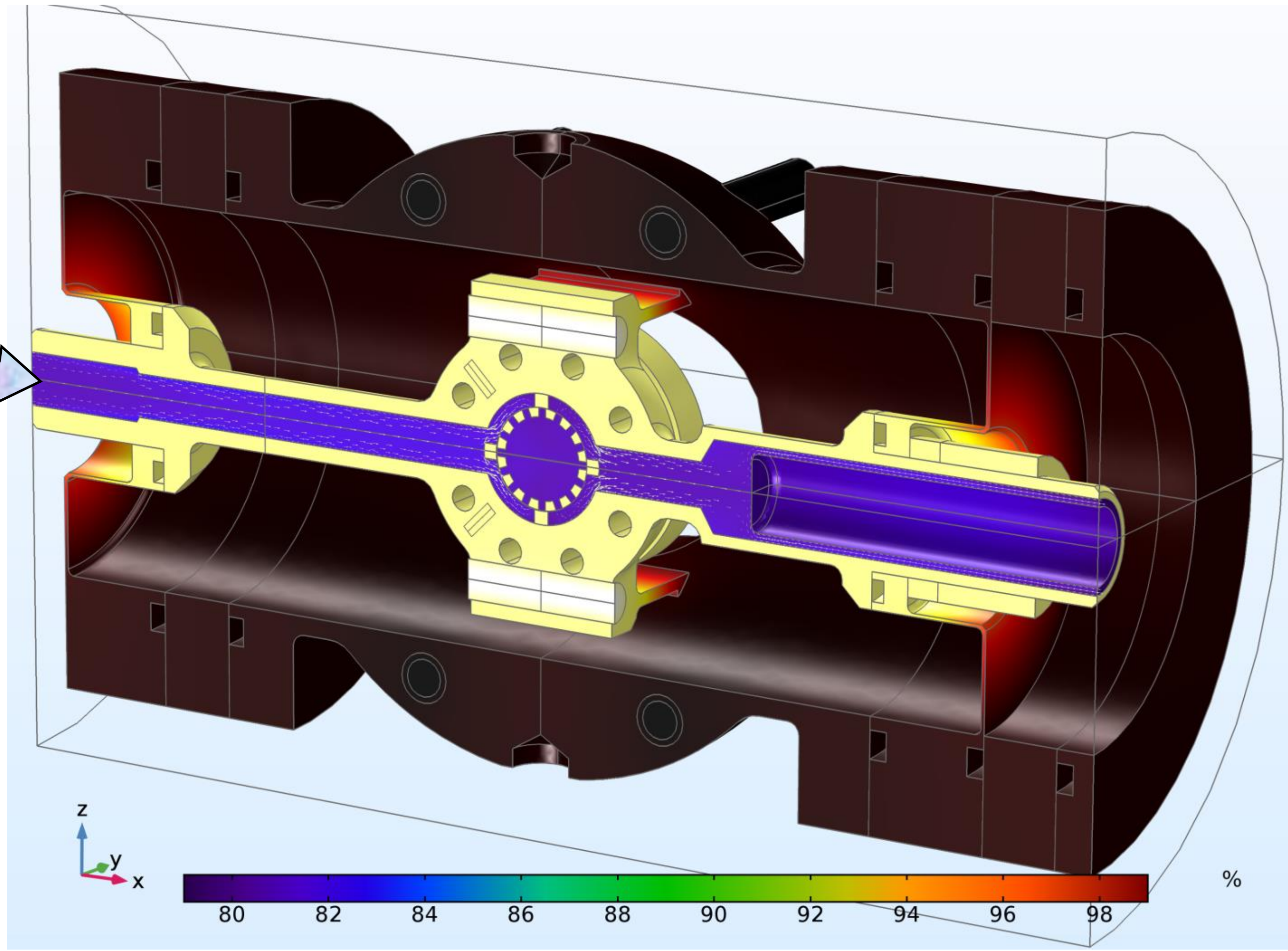
Σ ≈ 14 sec (dead time)

Climate response

❹ Change of climate

Response to a gas temperature jump at moist gas inlet

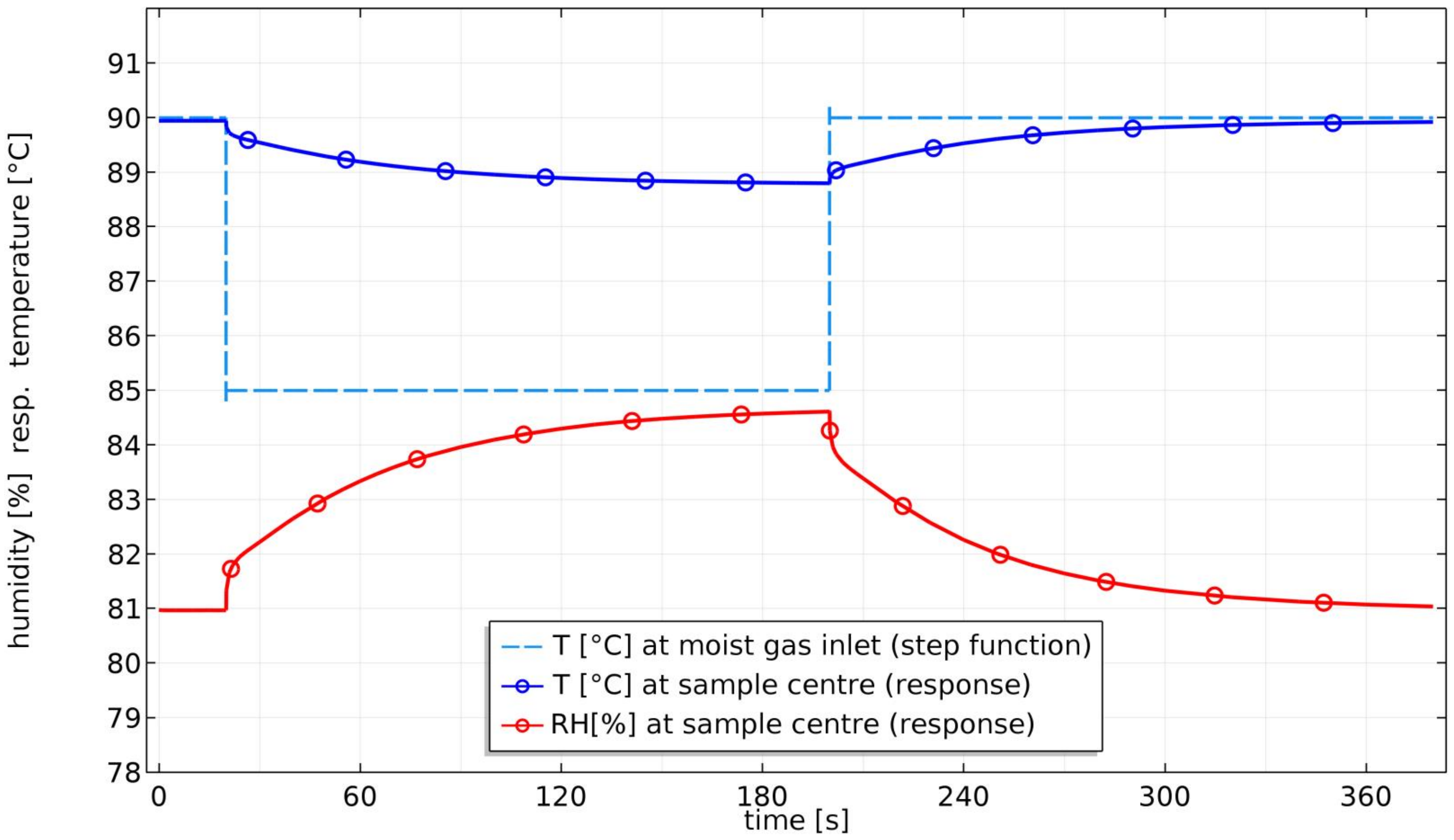
moist gas inlet



Climate response

❹ Change of climate

Response to a gas temperature jump at moist gas inlet

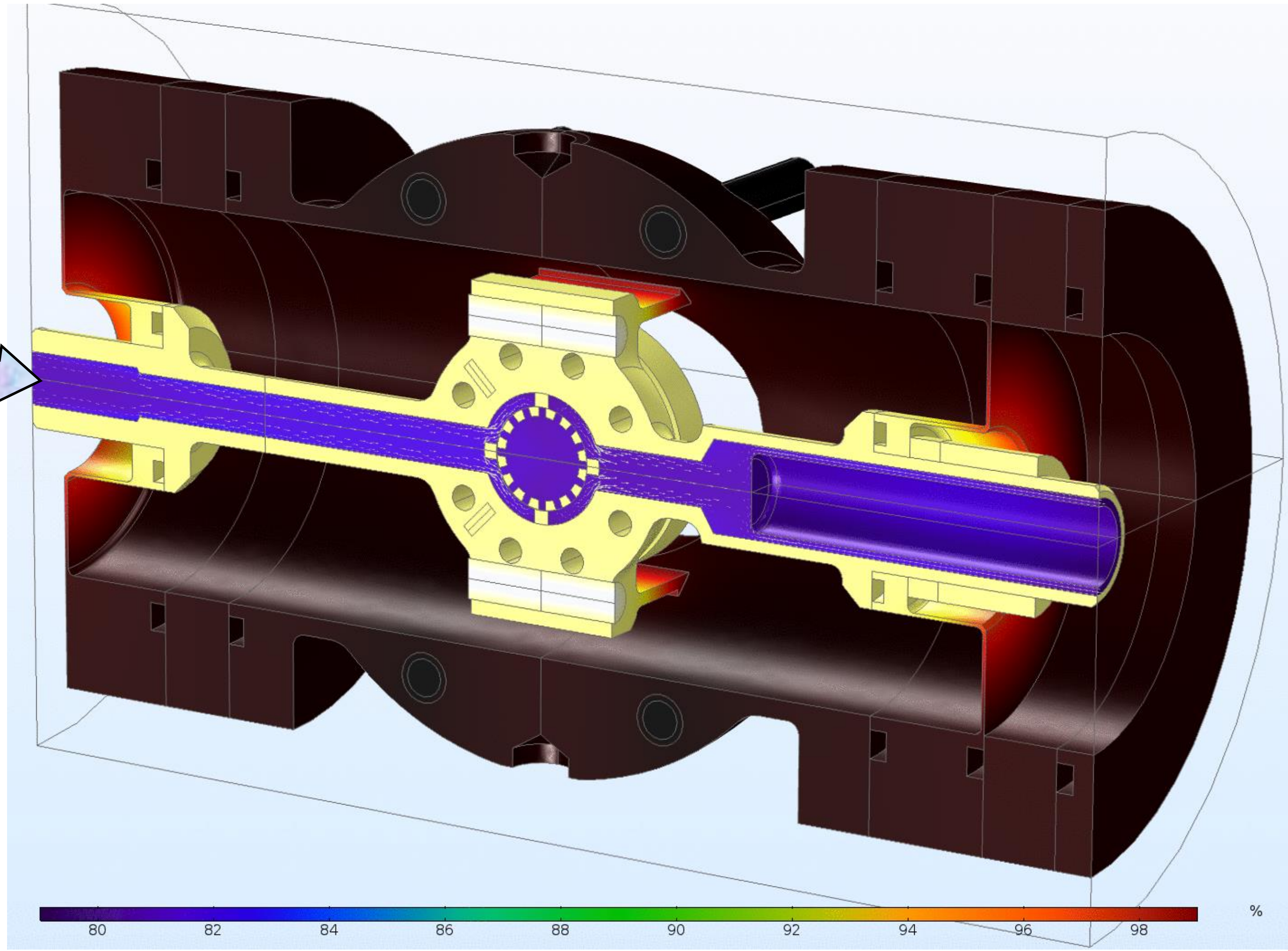


Humidity response

❹ Change of climate

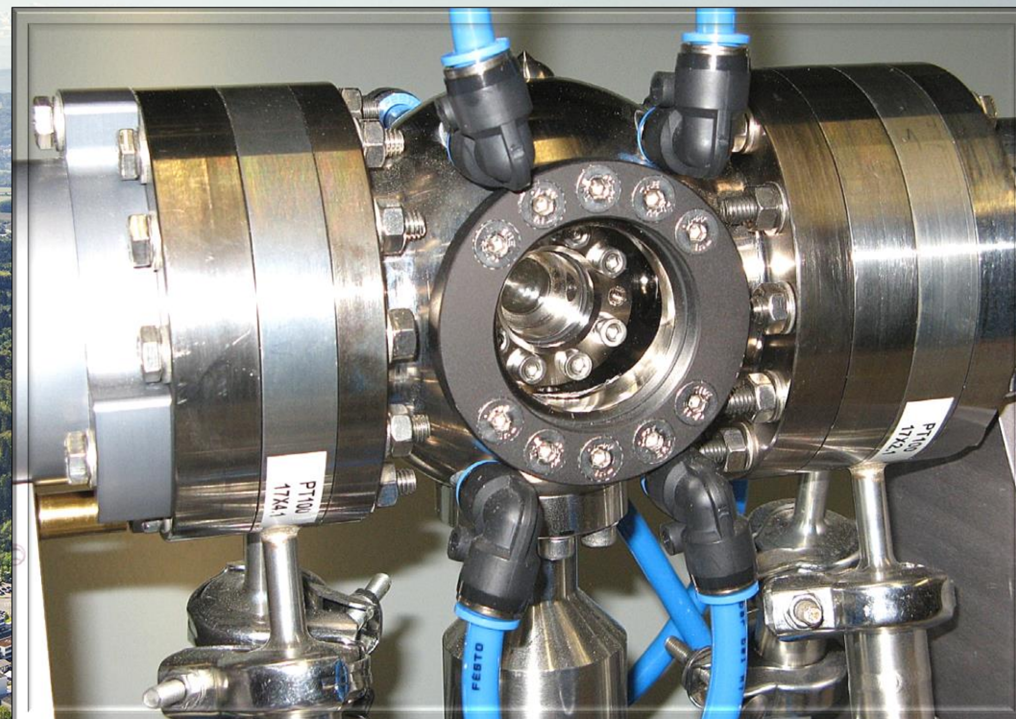
Humidity response to a gas temperature jump at moist gas inlet

T [°C] jump at gas inlet



We used CATIA, Mathcad and COMSOL Multiphysics® to design a climate chamber as sample environment. Design goals were, to understand and optimise

… temperature effects.
… humidity distribution.
… fluid flow.
… structural mechanics effects.



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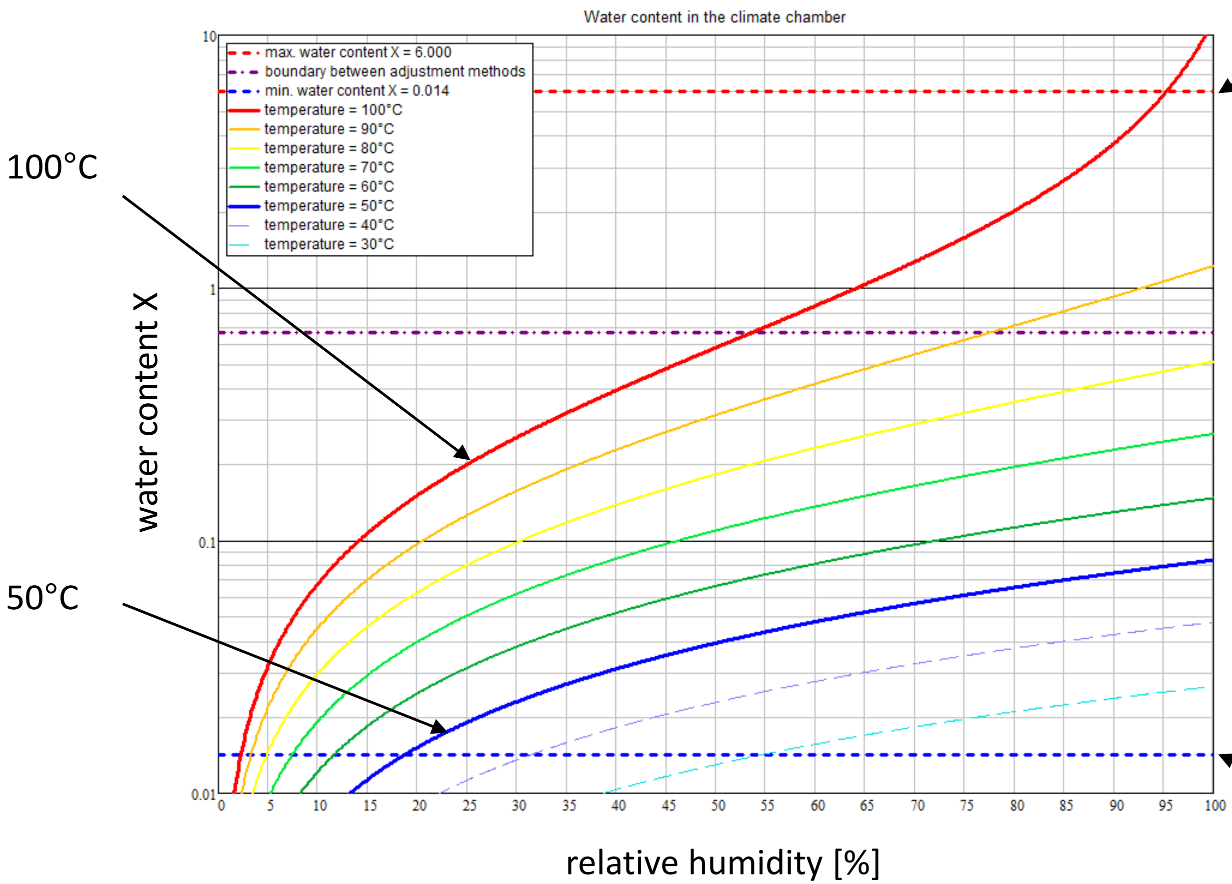
- Philipp Looser 3)

1) Electrochemistry Lab. (LEC)
2) Neutron Scattering Lab. (LNS)
3) Scientific Develop. Lab. (LDM)
4) PSI Mech. Prod. (AMI)

Appendix: Operation overview

Water content in the climate chamber



max. water content

$X_{CEM.max}$ = 6.000

$$\frac{X_{CEM.max}}{X_{CEM.min}} = 428$$

min. water content

$X_{CEM.min}$ = 0.014

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Appendix: Our Application

Small-angle Neutron Scattering (SANS)

The instrument :