EXPERIMENTAL TESTING OF BCV BENTONITE
WP HITEC AND GAS

JIŘÍ SVOBODA

CTU IN PRAGUE

CTU
CZECH TECHNICAL UNIVERSITY IN PRAGUE

eurad
European Joint Programme on Radioactive Waste Management

This project has received funding from the European Union's Horizon 20 research and innovation programme 2014-2018 under grant agreement N°847593

BCV

- BCV = Bentonite Černý Vrch
- Reference bentonite for research in Czech Republic
- Mg-Ca Bentonite

Wt.%	Anatase	Quartz	Montmorillonite	Mg-calcite	Goethite	Hematite	Kaolinite	Ankerite	Siderite	Illite
Original BCV	2.3	11.4	69.7	3.7	3.1	-	5	0.6	0.5	3.7

Wt.%	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MgO	MnO	CaO	Na_2O	K_2O	P_2O_5	F	CO_2	C	S	$H_2O(+)$	Total
BCV	51.86	2.34	15.56	11.41	0.14	2.82	0.2	2.83	0.37	1.02	0.51	0.12	1.68	0.17	< 0.010	9.06	100.09

	BCV_2017
Na^+ (%)	**11**
Ca^{2+} (%)	**23**
K^+ (%)	2
Mg^{2+} (%)	**64**
$CEC_{Cu\text{-}VIS}$ $(mmol^+ \cdot 100\ g^{-1})$	60.9

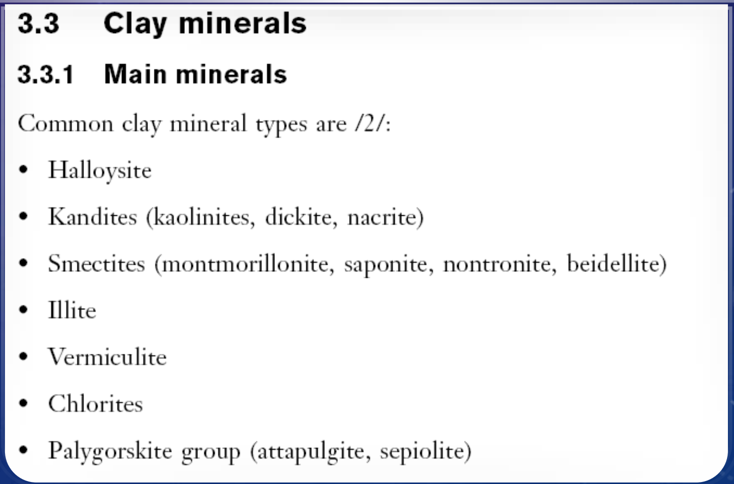
BENTONITE

- Bentonite is the name for a claystone which contains as main component the clay mineral Montmorillonite.
- The name bentonite comes from the „Fort Benton“ in the US state Wyoming, where geologists found at the end of the 19th century a plastic soil with unusual properties, which they called Bentonite.
- Bentonite was used already by the old indians as a kind of soap for washing their clothes.

MONTMORILLONITE

- Near the town Montmorillon (SW part of France) a plastic clay deposit had been discovered by French geologists, at the end of 19th century.
- Montmorillonite is the most important representative of the group of swellable three-layer minerals, which are called Smectites.
- The content of Montmorillonite is one of the most important quality parameter for raw bentonite as well as for processed bentonite products.

wikipedia



3.3 Clay minerals

3.3.1 Main minerals

Common clay mineral types are /2/:

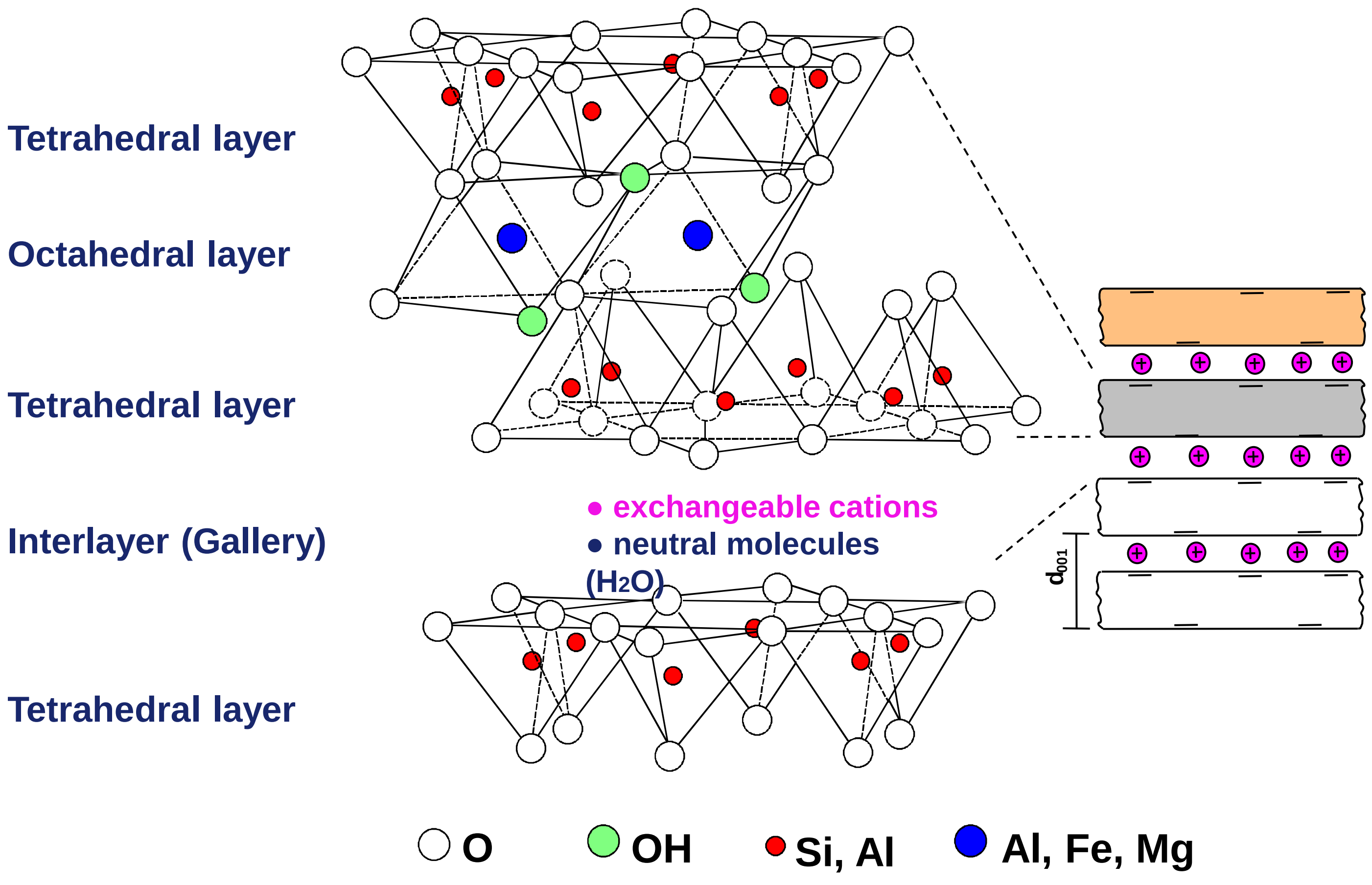
- Halloysite
- Kandites (kaolinites, dickite, nacrite)
- Smectites (montmorillonite, saponite, nontronite, beidellite)
- Illite
- Vermiculite
- Chlorites
- Palygorskite group (attapulgite, sepiolite)

SKB: Buffer and Backfill handbook

SMECTITE STRUCTURE

Li < Na < K < Ca < Mg < NH_4

”Ca/ Mg/ Na bentonite”

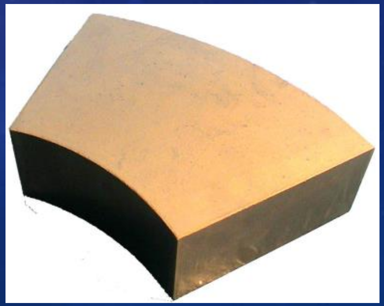


Dietrich Koch, S&B, ABM Projectmeeting, Äspö, 2006-11-30

BENTONITE

- Naturally occurring material → Inhomogeneities → Uncertainty/**Natural spread** in material properties, accessory minerals – ***known unknown***
- Industrially processed for most needs (including DGR)
- Various forms
  - Natural form
  - Processed
    - **Powder**
    - **Pellets**
    - **Compacted blocks**

SKB: TR-17-06



ROLE OF BENTONITE IN EBS

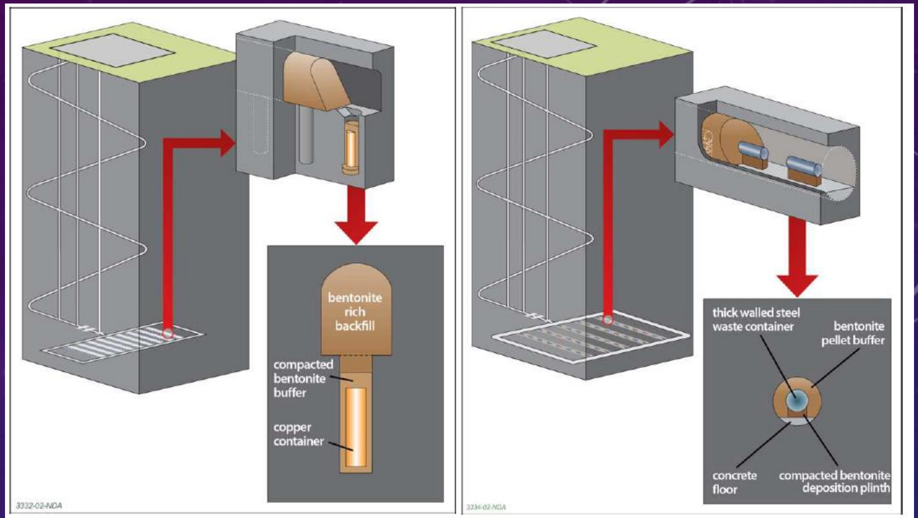


Figure 2-26. RWM illustrative designs for a higher strength rock (based upon KBS-3V, left) and lower strength sedimentary rock type (based upon NAGRA concept, right) after RWM (2016)

Bentonite is main material of buffer and backfill

- Buffer – surrounds waste package
  - Waste package protection (from host rock movements,…)
  - Isolation of waste (physical, hydraulic, chemical,…)
  - Minimise radionuclide release to environment (limit water movement, sorption,…)
  - Heat transfer
- Backfill – (back)filling of all empty spaces in DGR (galleries, tunnels, shafts,…)
  - Hydraulic isolation of EBS system
  - Support for backfill

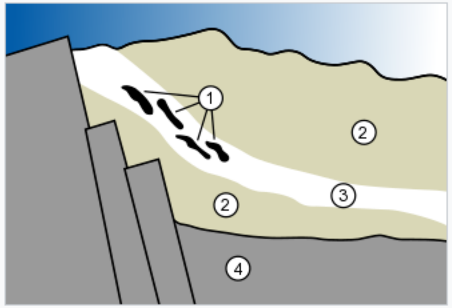
Requirements on properties of EBS system/materials → **bentonite**

REQUIREMENTS ON BENTONITE

- **Long-term stability** — **- properties shall be predictable for the lifetime of repository**

  Hint: Natural analogues
- Extremely low permeability — - limitation of water movement (corrosion, pollutant transfer)
- Extremely high plasticity — - mechanical protection and sealing
- Swelling — - sealing
- Self-healing — - sealing, recovery after damage
- High thermal conductivity — - cooling of waste package
- …

Note: Performance of bentonite (properties) depend on density and water content



Geological situation in Gabon leading to natural nuclear fission reactors

1. Nuclear reactor zones
2. Sandstone
3. Uranium ore layer
4. Granite

wikipedia

EBS ERECTION

Bentonite has to be emplaced – technological process

→ Unknowns due to technology/installation of EBS

- Gaps/joints between blocks & layers
- Free space between pellets

- Unfilled voids (or less material) due to technological reasons
  - Space for tools and manipulation
  - Emplacement accuracy
  - Tolerances/Uneven surfaces
  - Too small space to access/fill
  - Errors…

Note: the installation method has influence on average density of emplaced component

SKB: TR-17-06

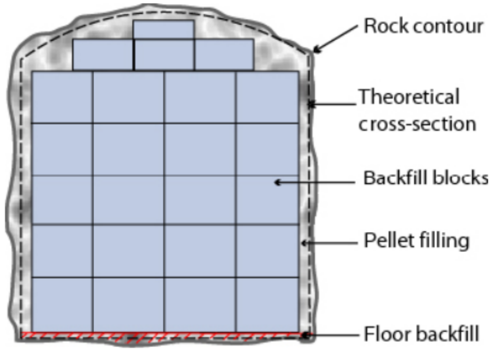


Figure 1-2. Cross section of a backfilled KBS-3V deposition tunnel showing the three main components of the backfill. 1) precompacted blocks, 2) pellet fill and 3) material placed under the blocks to provide stable foundation for the blocks (Keto et al. 2009a).

Figure 2-12. Placement trials of tunnel fill using twin-auger technique. (De Bock et al. 2008: Note NAGRA canister-sized cylinder placed in tunnel.)

SKB: P-11-44

WHAT SHOULD WE TEST AND WHY?

- Material properties and composition
  - Density dependent
  - Water content dependent
- System properties
  - Heterogenous materials
  - Material in various form in one system
  - Discontinuities, gaps, …

- Influence of:
  - Temperature
  - Water (flow, composition,…)
  - Disturbing events (gas breakthrough, seismic activity,…)
  - …

GAS

MECHANISTIC UNDERSTANDING OF GAS TRANSPORT IN CLAY MATERIALS

GAS – TYPICAL AND ALTERNATIVE APPROACH (DILATANT FLOW/FRACTURATION)

- Typical - Gas breakthrough test
  - Slow injection of gas until the pathway is created
  - Slow increase of gas pressure
  - Pressure at gas breakthrough obtained
  - Very, very slow…

- Alternative - Fast gas breakthrough test
  - High gas pressure applied **immediately**
  - Time to breakthrough measured
  - **Fast test. Easy repetition.**
  - Gas breakthrough pressure NOT obtained
  - Qualitative result (in terms of breakthrough event)

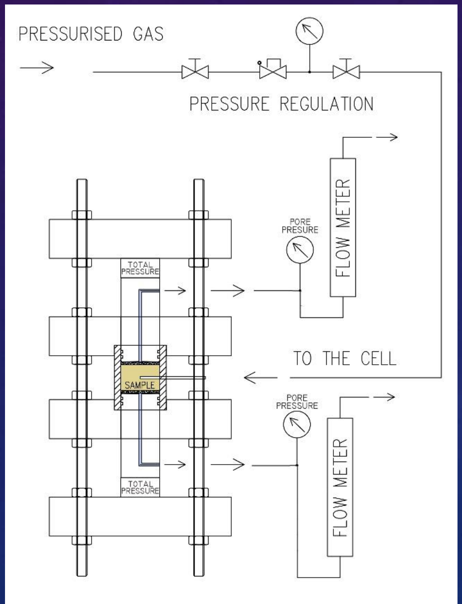
WHAT WE WANT TO KNOW?

- At what pressure does a breakthrough happen
- What happens to the EBS performance
- What are the influences of material density, heterogeneity, discontinuities,…

WP GAS - CTU OVERVIEW

Task 2 – "Slow"

- Material: BCV - homogeneous samples
- Permeameter: hydraulic cond., swell. pressure
- Long-term air injection tests – via injection needle or sintered steel plates
- Incremental pressure increase until breaktrough



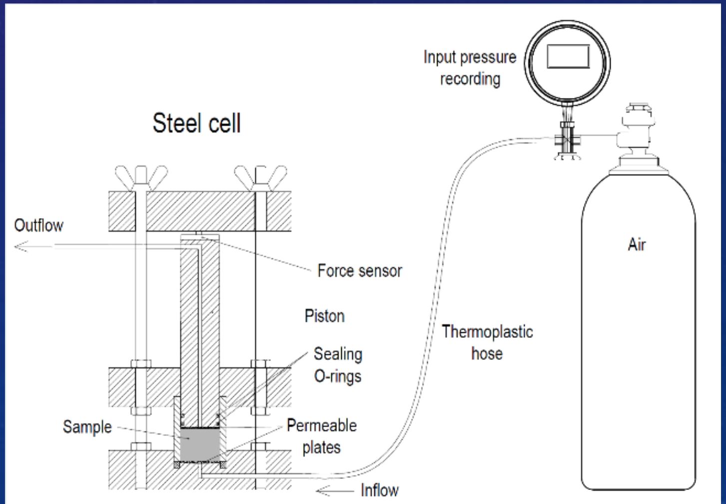
Air injection system

Permeameter

Task 3 – "Fast" and repeated

- Material: BCV - homogeneous and inhomogeneous samples
- Permeameter: hydraulic cond., swell. pressure
- Short-term air injection tests, high pressures
- Repeated cycles of gas injection and resaturation

Air injection system



SLOW TESTS

T2

A TALE OF LOST NEEDLE…

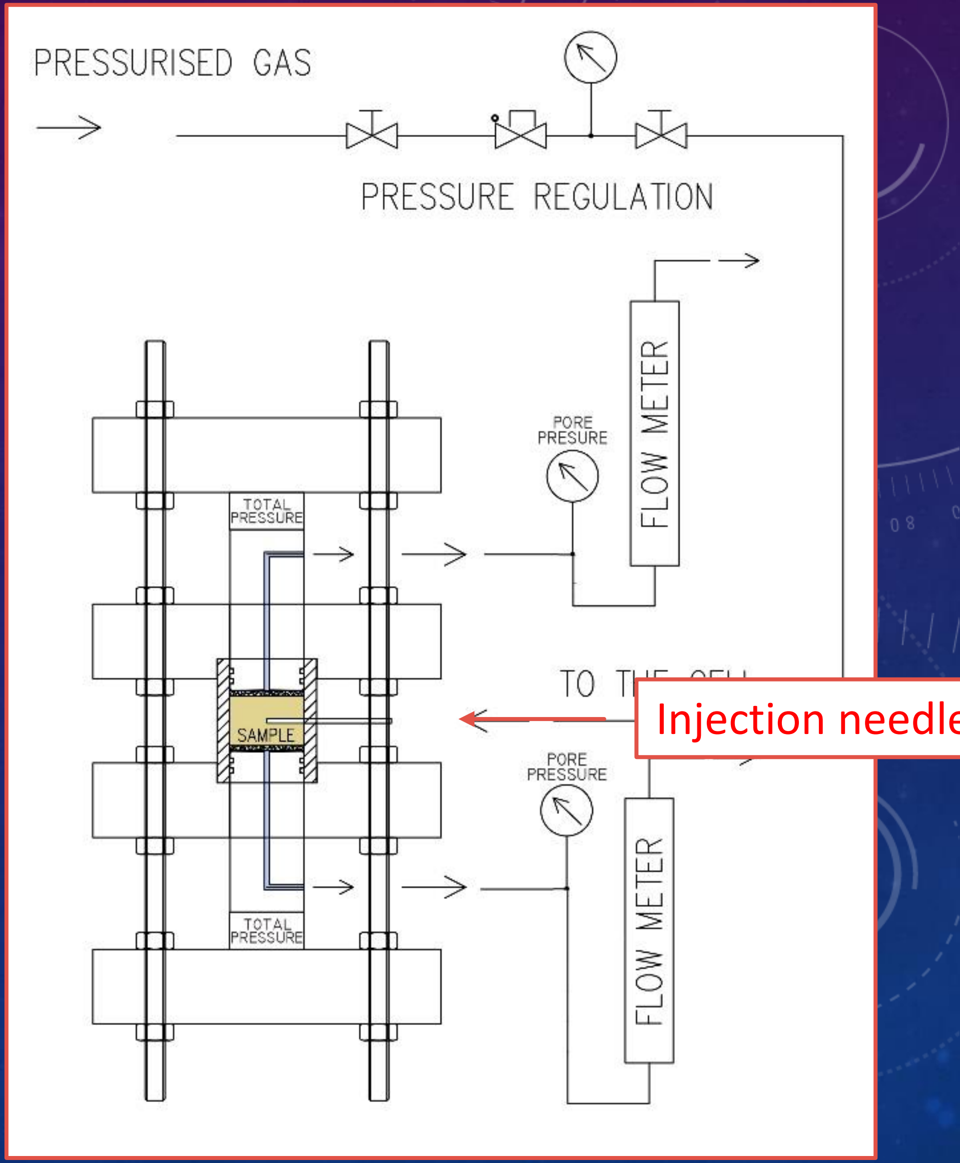
The first idea was to inject gas into centre of sample via needle and try measure the desaturation (water outflow).

6 months of sample saturation, then test. It didn’t work out 3 times…

- Port leaked
- Gas escaped around needle
- Needle corroded out
- Outflow (water) measurement not sensitive enough

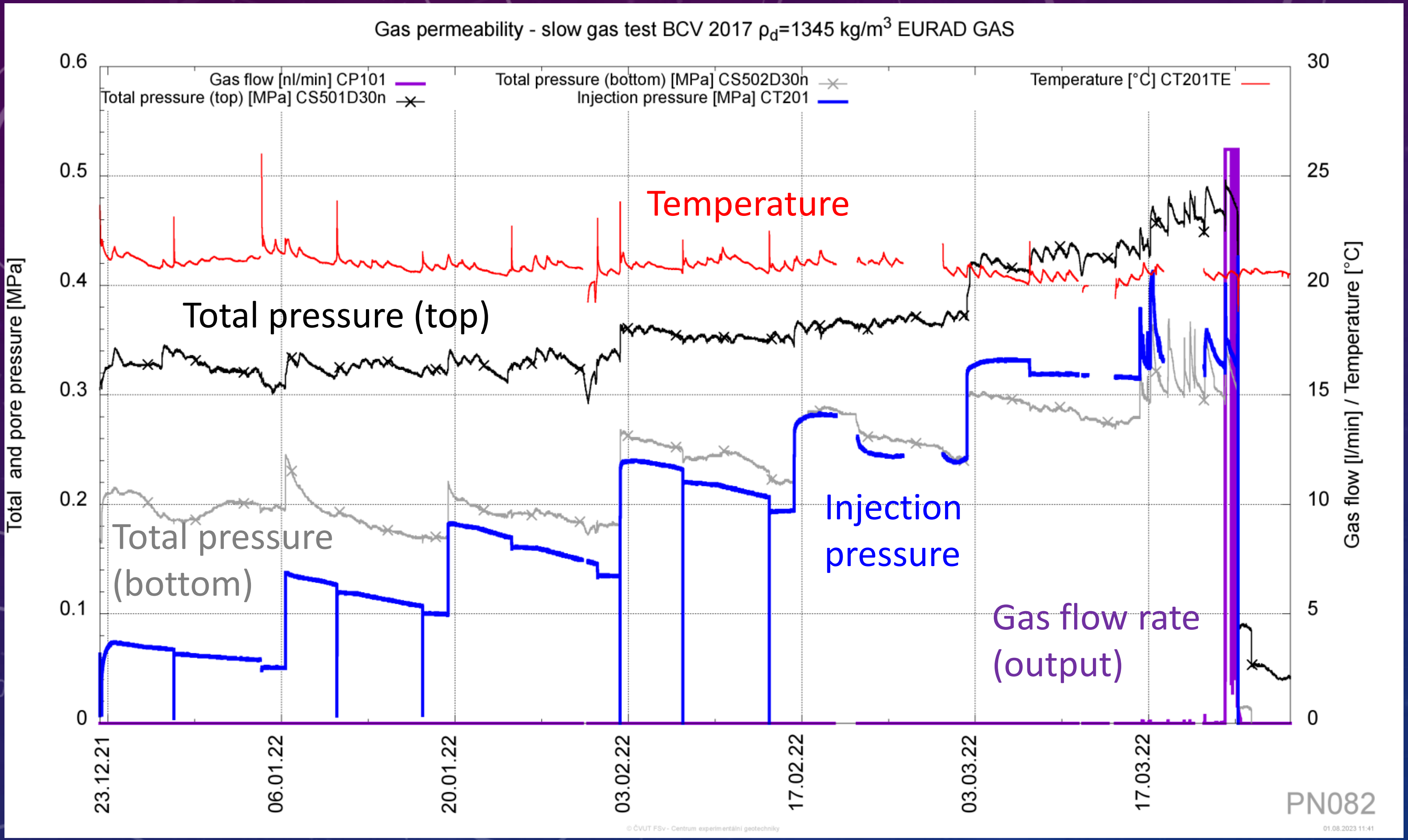
→ Time for Plan B

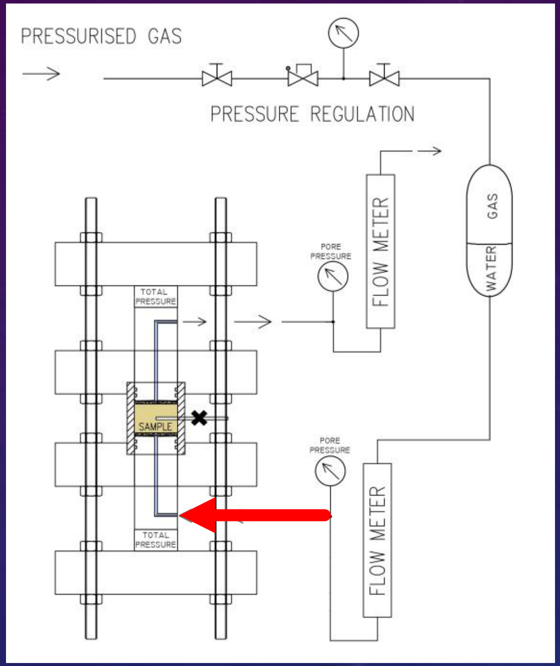
- Additional cell
- Test from bottom
- Improved setup – replacement of needle by PTFE tube



sample no.	target ρ_d [kg/m^3]	preparation of sample	saturation/resaturation phase [days]	plan of gas pressure test	gas test no.	start of the gas pressure test	end of the gas pressure test	note	gas injection point	total pressure - top sensor [MPa]	total pressure - bottom sensor [MPa]	initial injection pressure - in first step [MPa]	loading step [kPa]/time [days]	duration of gas injection [days]	breakthrough pressure [MPa]
P766	1300	20.08.2020	168	12.02.2021	PN069	26.04.2021	26.04.2021	**unsuccessful test** - technical problems with needle at the centre of the sample	Injection needle	0,45	-	0,6	50/14	0,5	---
P805	1345	10.05.2021	168	26.10.2021	PN077	26.10.2021	26.10.2021	**unsuccessful test** - gas passes through testing cell, technical problems with the injection needle	Injection needle	0,40	-	0,2	50/14	5	---
			50	22.12.2021	PN082	22.12.2021	24.03.2022	resaturation of the sample after unsuccessful test	to base	0.38	0,18	0,07	50/14	84	**0,37**
P815	1394	06.09.2021	93	14.03.2022	PN081	14.03.2022	14.06.2022	**unsuccessful test** - technical problems with gas leakage during the test	Injection needle	2,21	1,14	0,38	50/14 than 50/7 (after 3rd step)	98	**0,84**
			80	15.09.2022	PN092	23.09.2022	14.03.2023		to base	2,03	1,10	0,57	50/7	172	**2,5**
P823	1473	09.11.2021	168	26.04.2022	PN086	26.04.2022	14.03.2023	simple measuring apparatus (with one piston) and with gas injection to the base of the sample	to base	3,00	-	1,54	50/7	322	**4,43**
P840	1500	20.04.2022	168	07.10.2022	PN107	21.03.2023	11.07.2023	1st step - 2.35 MPa	Injection needle	3,76	2,74	2,35	50/7	110	**3,26**

BCV 1345 – TEST PN082



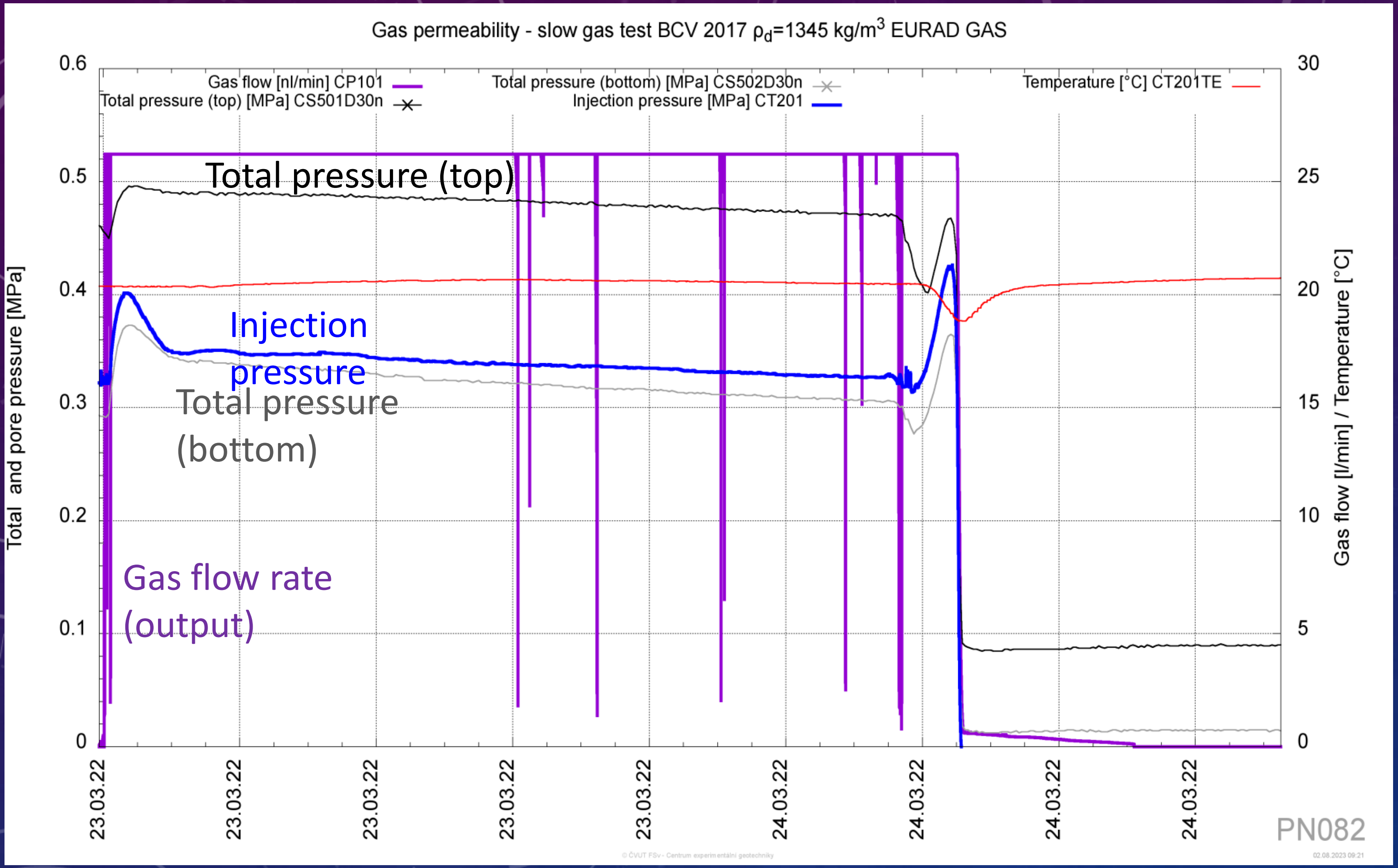


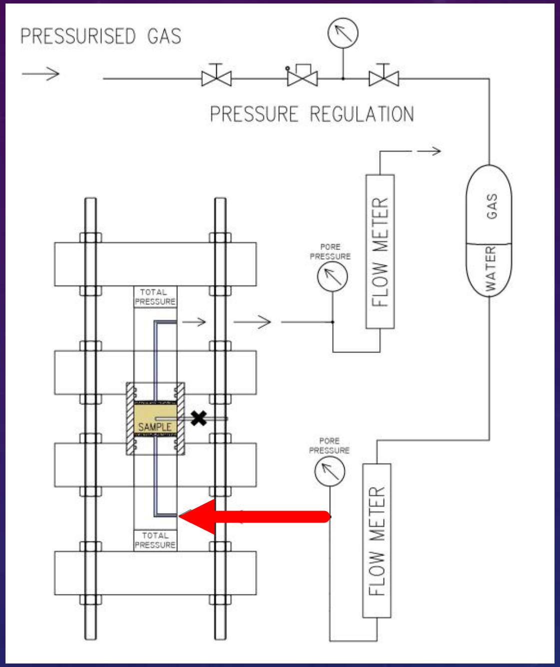
Saturation: 168 + 50 days
Total (swelling) pressure:
Top sensor 0.38 MPa
Bottom sensor 0.18 MPa

Initial pressure step: 0.07 MPa
Pressure increments:
50 kPa (14 days)
Breaktrough pressure: 0.37 MPa
Theoretical swelling pressure:
1.5 – 2.1 MPa for 1400 kg/m^3

Start: 22-12-2021 End: 24-03-2022

BCV 1345 – TEST PN082 _BT EPISODE





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Bottom sensor 0.18 MPa

Initial pressure step: 0.07 MPa
Pressure increments:
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Breaktrough pressure: 0.37 MPa
Theoretical swelling pressure:
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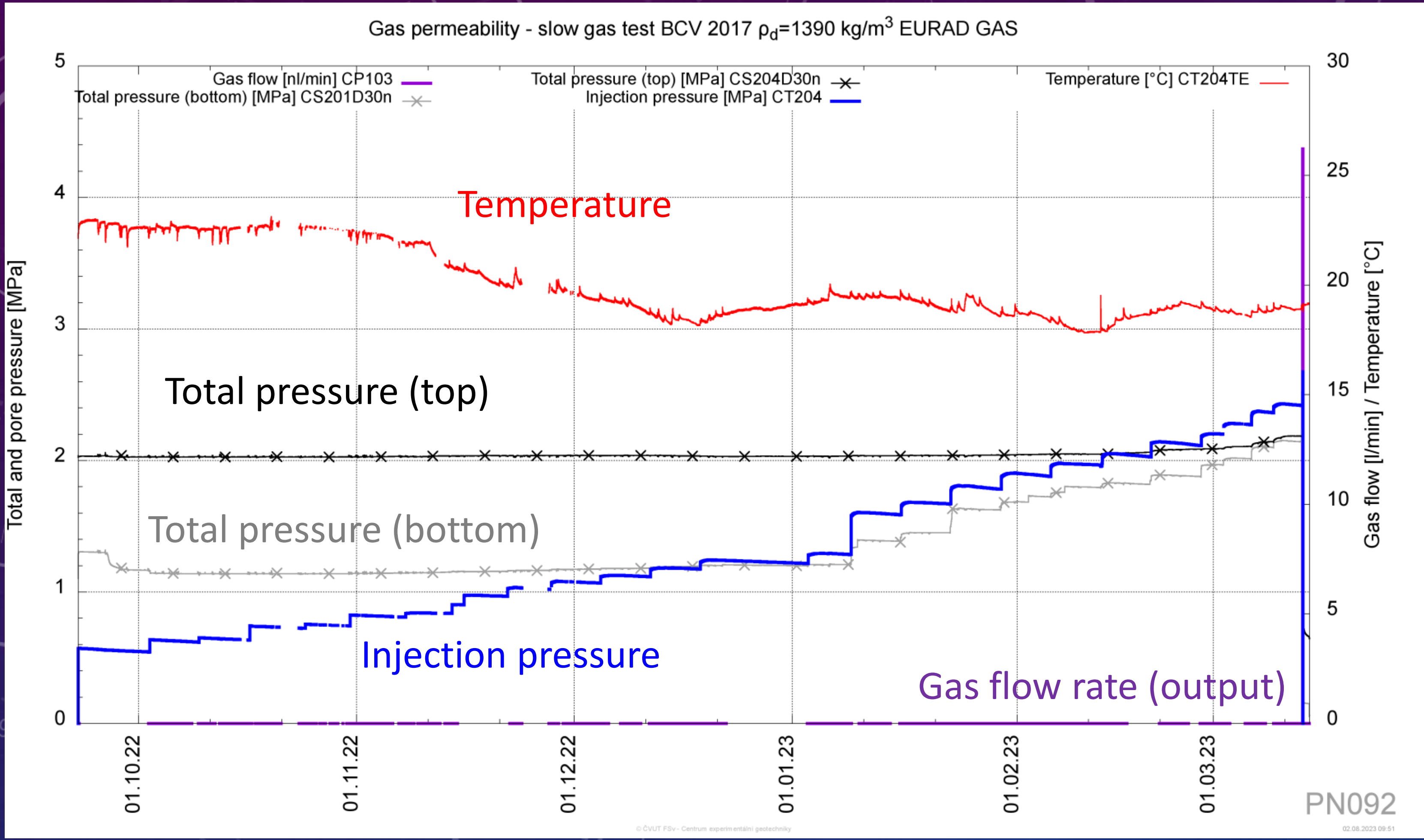
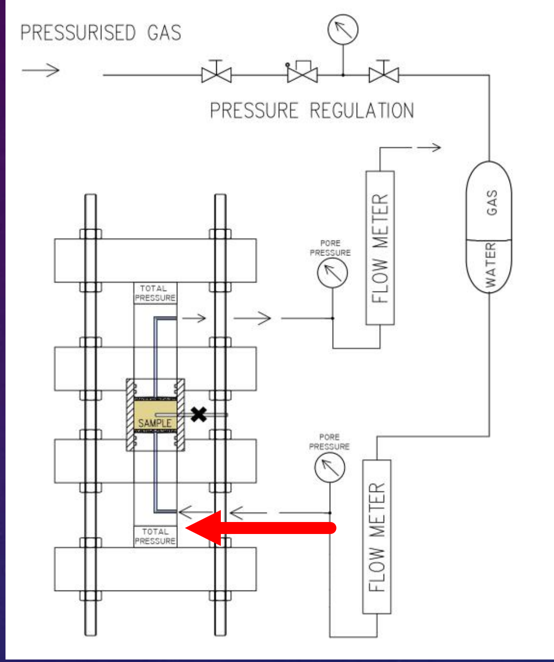
Start: 22-12-2021 End: 24-03-2022

BCV 1395 – TEST PN092





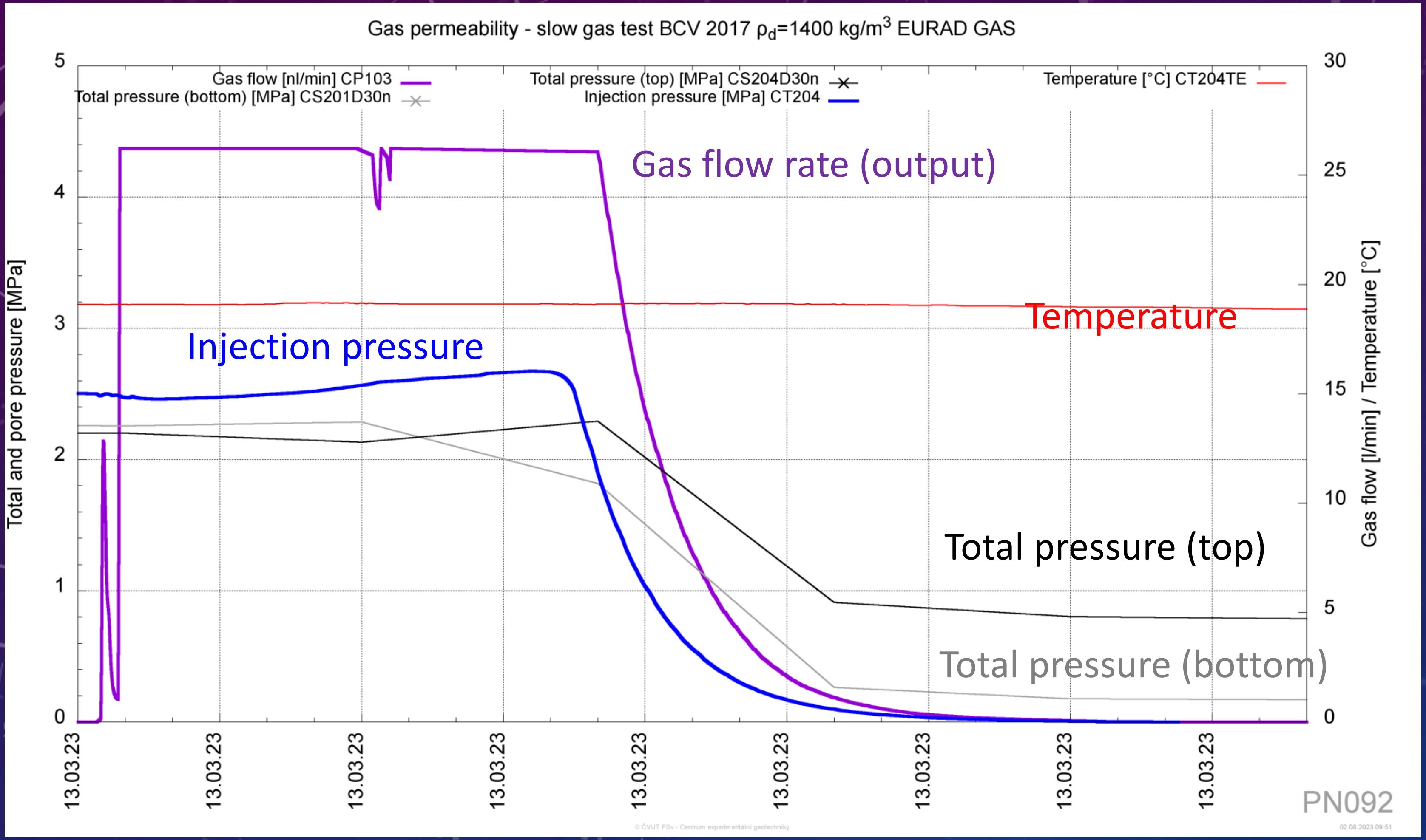
Start: 23-09-2022 End: 14-03-2023

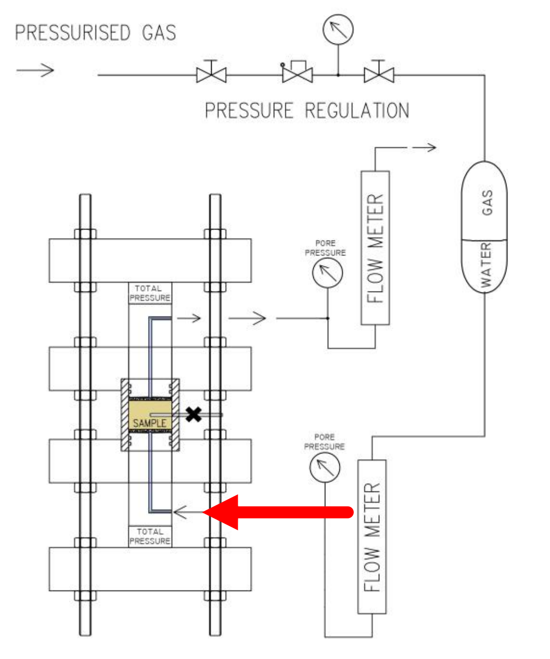
Initial saturation: 80 days
Total (swelling) pressure:
Top sensor 2.03 MPa
Bottom sensor 1.10 MPa

Initial pressure step: 0.6 MPa
Pressure increments:
50 kPa (7 days)
Breaktrough pressure: 2.5 Mpa

Theoretical swelling pressure: 1.5 – 2.1 MPa for 1400 kg/m^3

BCV 1395 – TEST PN092 - BT EPISODE





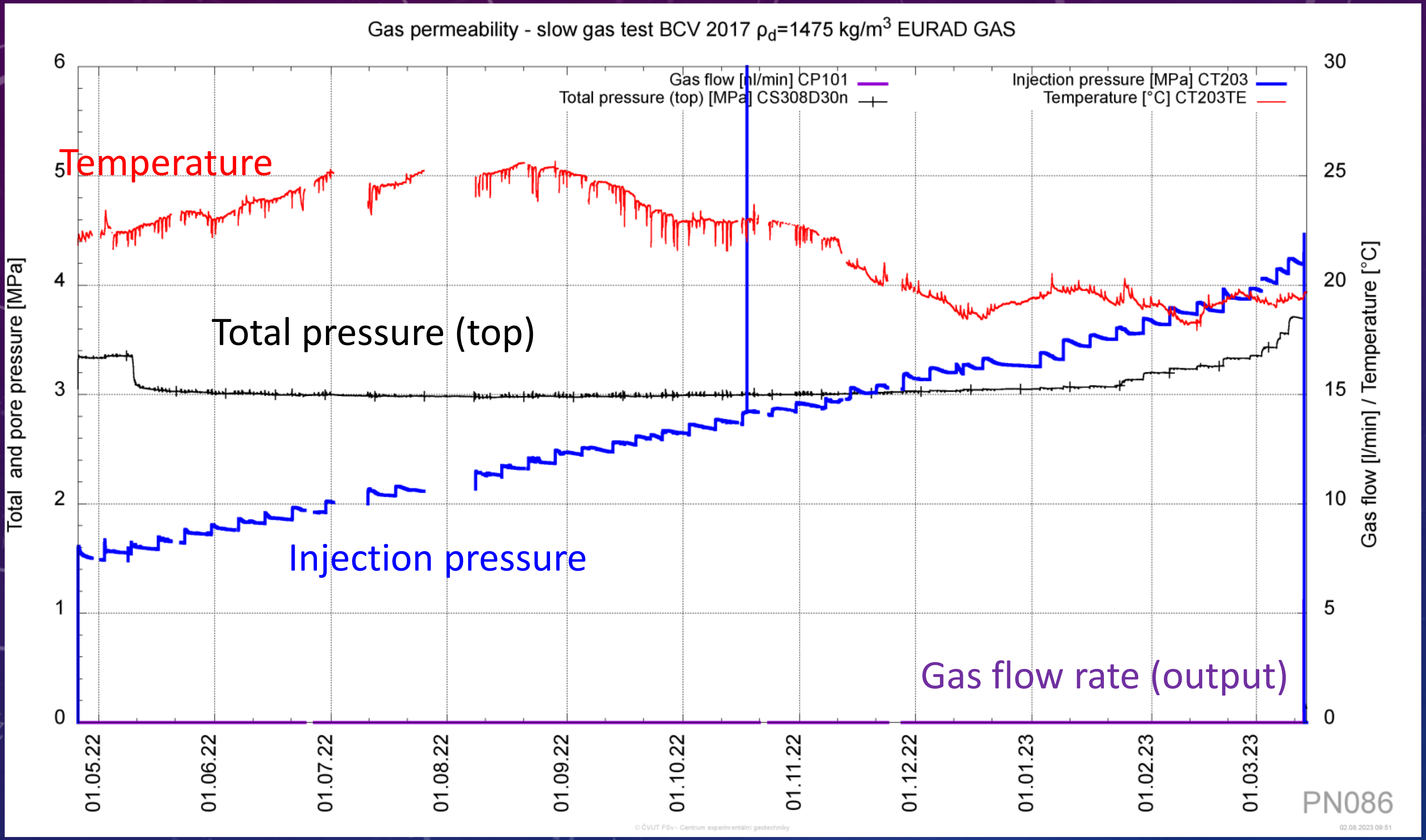
Initial saturation: 80 days
Total (swelling) pressure:
Top sensor 2.03 MPa
Bottom sensor 1.10 MPa

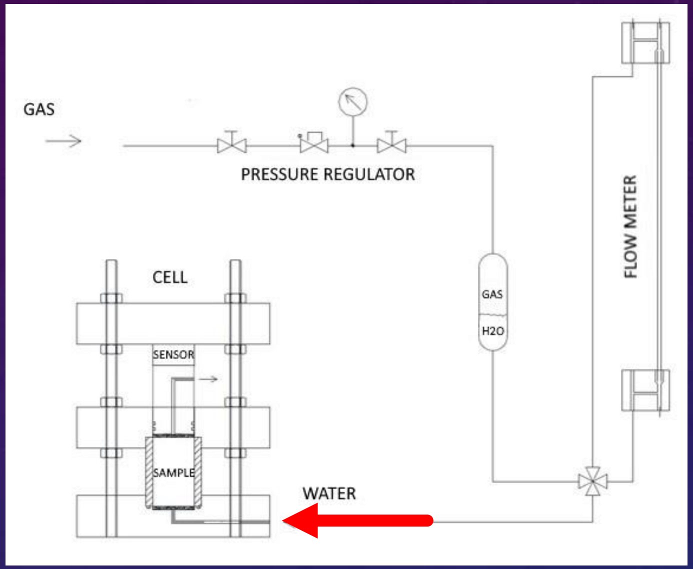
Initial pressure step: 0.6 MPa
Pressure increments:
50 kPa (7 days)
Breaktrough pressure: 2.5 MPa

Theoretical swelling pressure: 1.5 – 2.1 MPa for 1400 kg/m^3

Start: 23-09-2022 End: 14-03-2023

BCV 1475 – TEST PN086





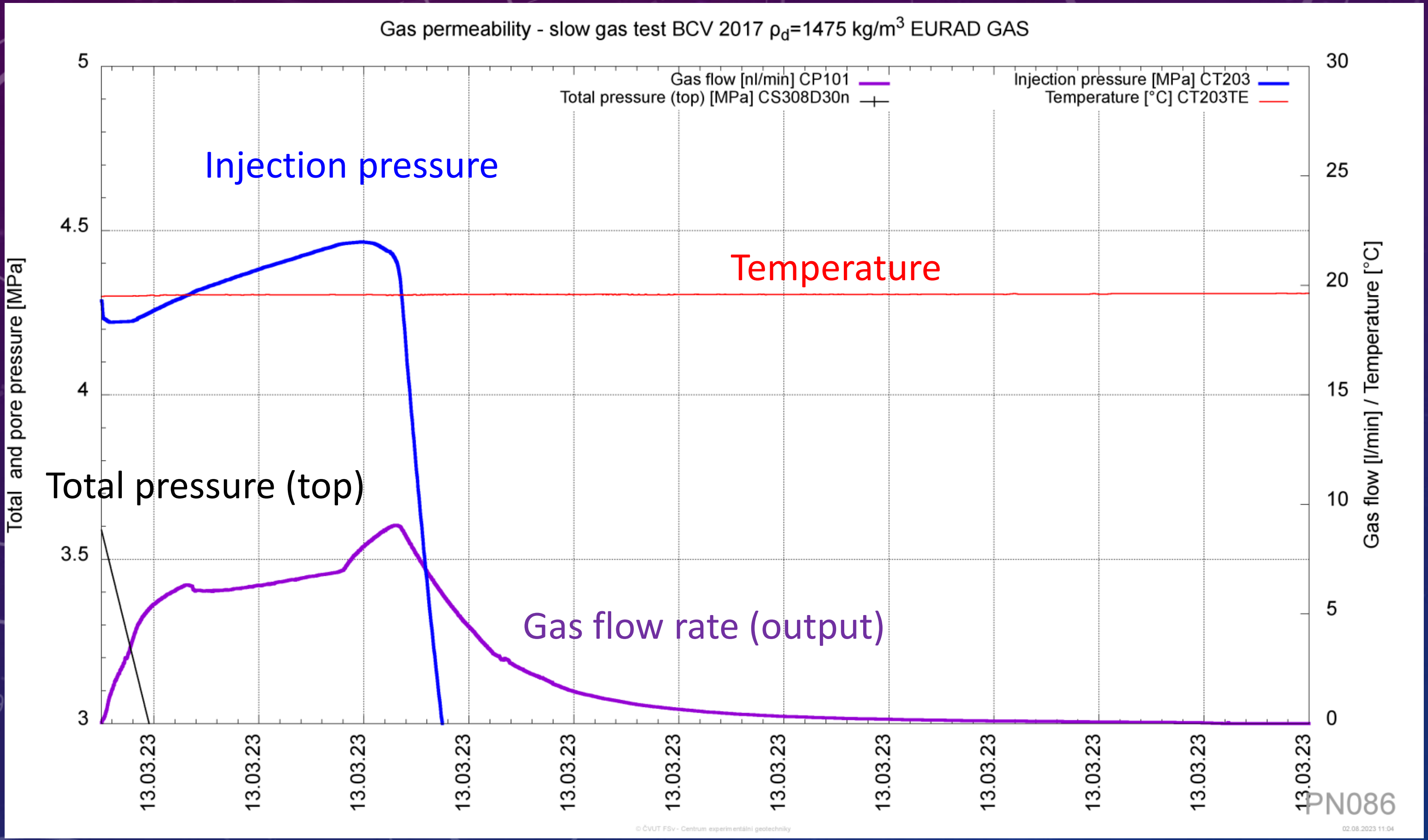
Initial saturation: 168 days
Total (swelling) pressure:
Top sensor 3.00 MPa

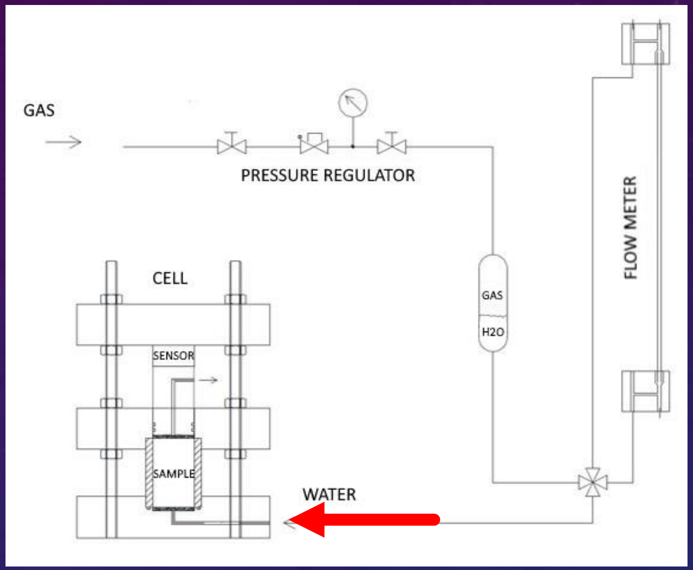
Initial pressure step: 1.5 MPa
Pressure increments:
50 kPa (7 days)
Breakthrough pressure: 4.43 MPa

Theoretical swelling pressure: 2.1 – 3.0 MPa for 1450 kg/m^3

Start: 26-04-2022 End: 14-03-2023

BCV 1475 – TEST PN086 – BT EPISODE





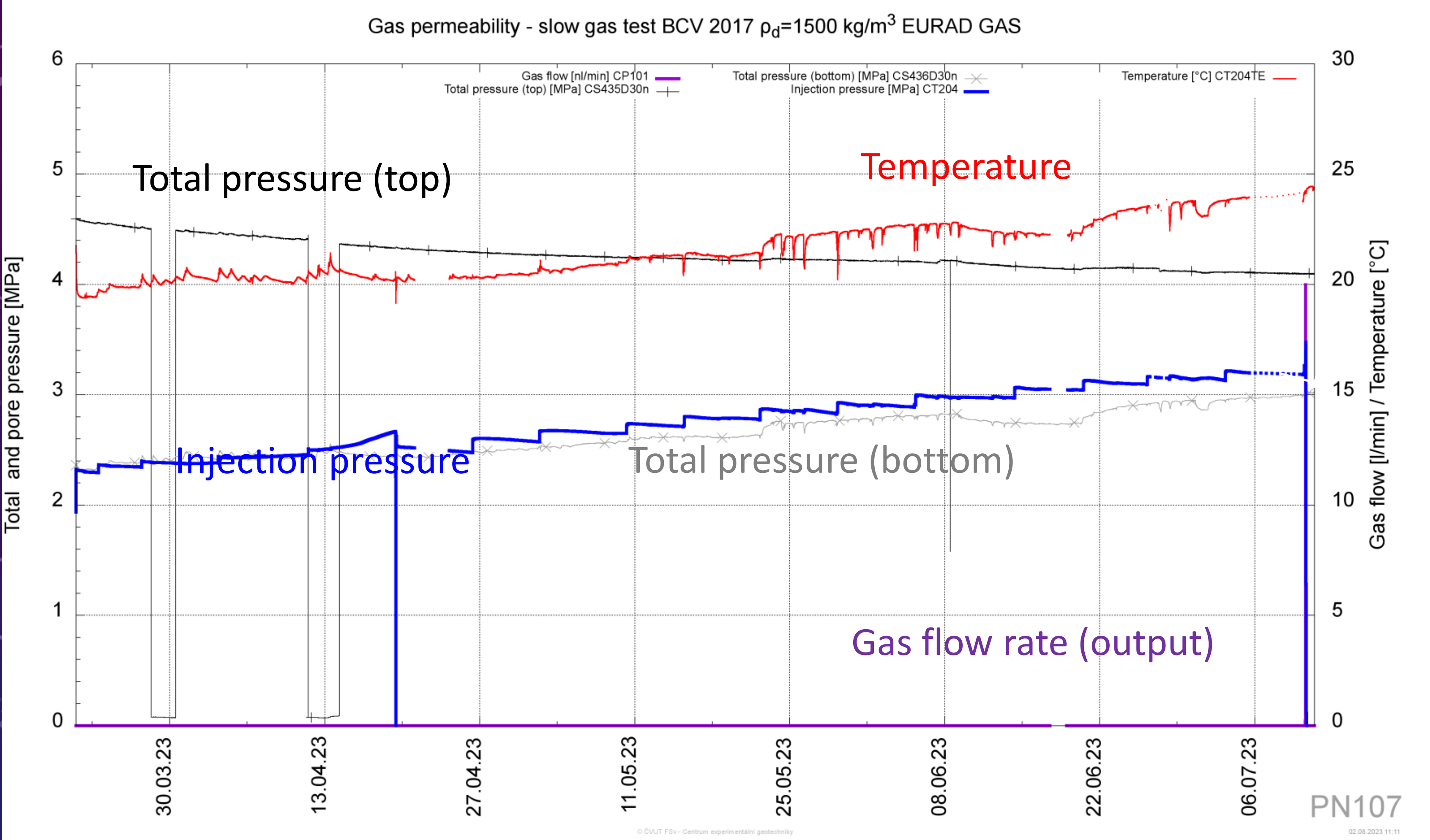
Initial saturation: 168 days
Total (swelling) pressure:
Top sensor 3.00 MPa

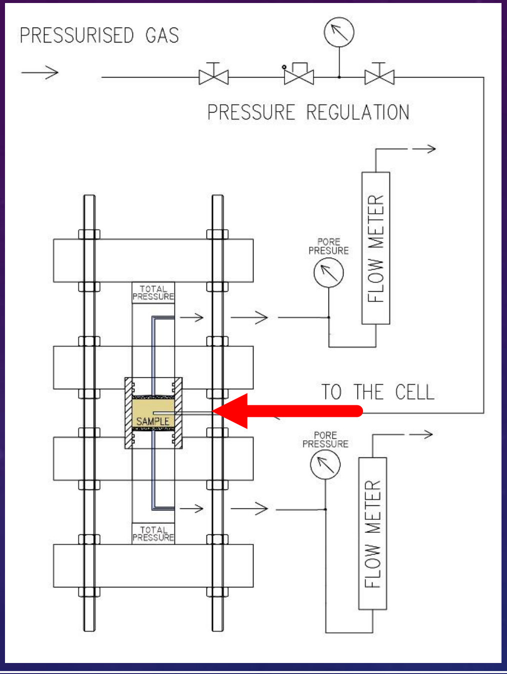
Initial pressure step: 1.5 MPa
Pressure increments:
50 kPa (7 days)
Breaktrough pressure: 4.43 MPa

Theoretical swelling pressure: 2.1 – 3.0 MPa for 1450 kg/m^3

Start: 26-04-2022 End: 14-03-2023

BCV 1500 – TEST PN107



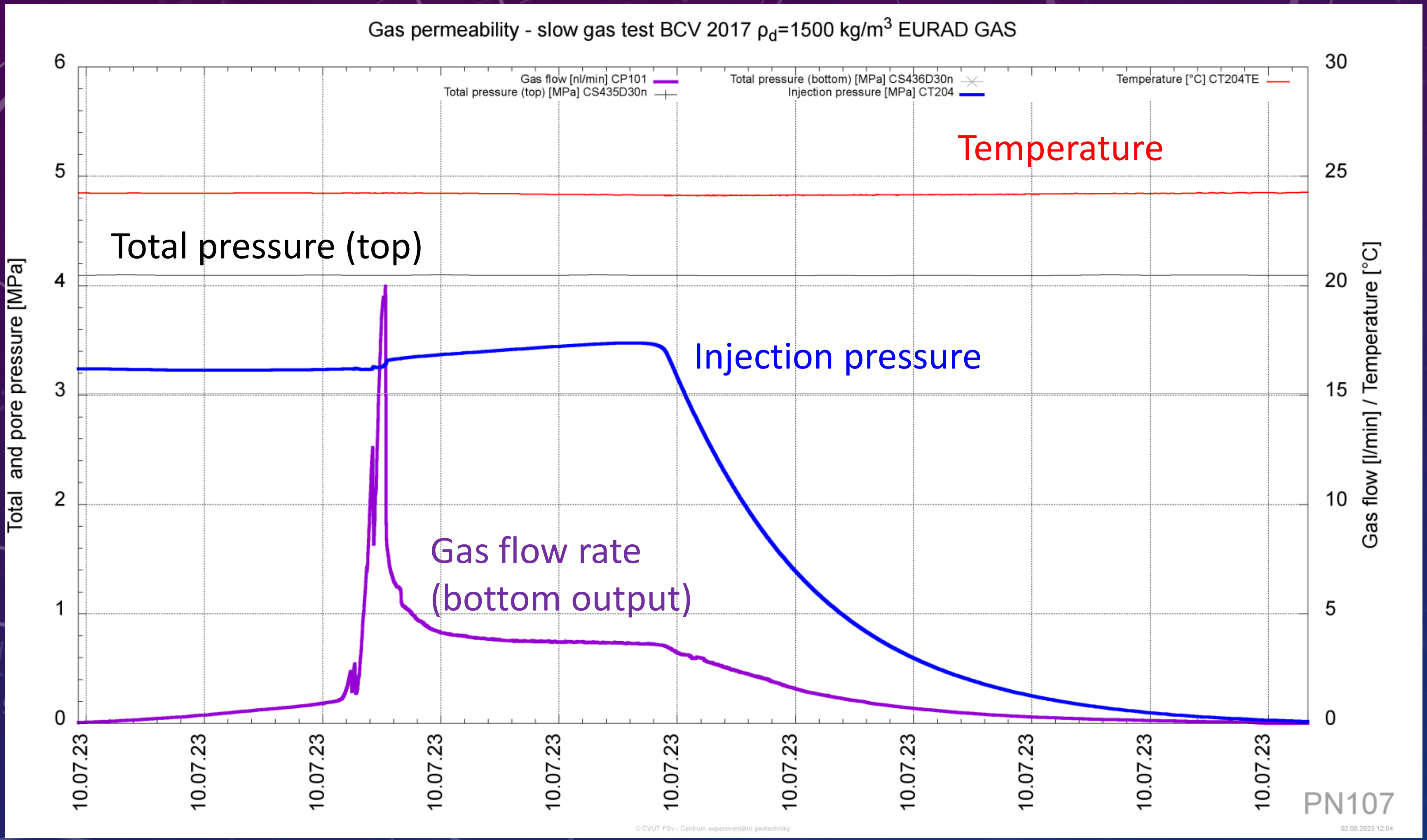


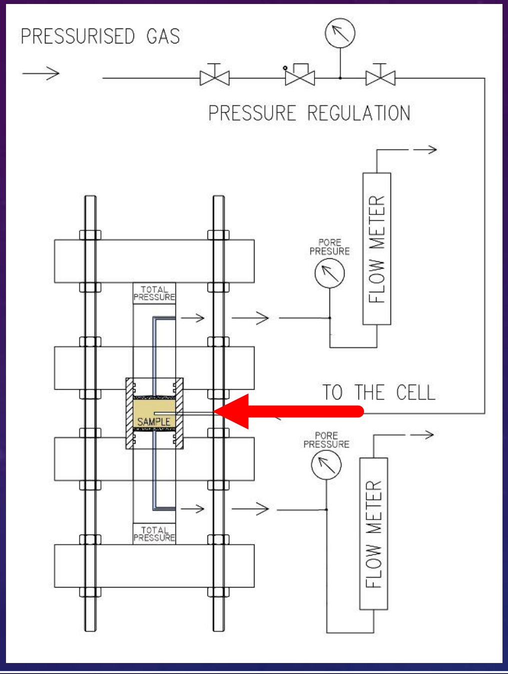
Saturation: 168 days
Total (swelling) pressure:
Top sensor 3.76 MPa
Bottom sensor 2.74 MPa

Initial pressure step: **2.35** MPa
Pressure increments:
50 kPa (7 days)
Breakthrough pressure:
3.26 MPa
Theoretical swelling pressure:
1.9 – 5.2 MPa for 1500 kg/m^3

Start: 21-03-2023 End: 11-07-2023

BCV 1500 – TEST PN107 – BT EPISODE





Saturation: 168 days
Total (swelling) pressure:
Top sensor 3.76 MPa
Bottom sensor 2.74 MPa

Initial pressure step: **2.35** MPa
Pressure increments:
50 kPa (7 days)
Breaktrough pressure: 3.26 MPa
Theoretical swelling pressure: 1.9 – 5.2 MPa for 1500 kg/m^3

Start: 21-03-2023 End: 11-07-2023

CT SCAN P840

Scene coordinate system
5.1225 mm
Top 1
5 mm
33%
Scene coordinate system
1.0826 mm
Right 1
4 mm
46%
Scene coordinate system
-4.8457 mm
Front 1
3.5 mm
47%
Scene coordinate system
3D

SLOW TESTS - CONCLUSION

A lot of technical problems…

Tests with gas injection into the base of the cylindrical sample

- The total pressure sensors react to the injection pressure – mechanical behaviour of the sample – a combination of the „plastic“ state of the sample and friction
- The breakthrough events registered for values of pressures above the swelling pressure

Tests with injection needle

- The breakthrough events registered for values of pressures above the swelling pressure

Air vs Hydrogen

- The results of test with air are giving similar results to tests with hydrogen (tests by UJV)

FAST TESTS

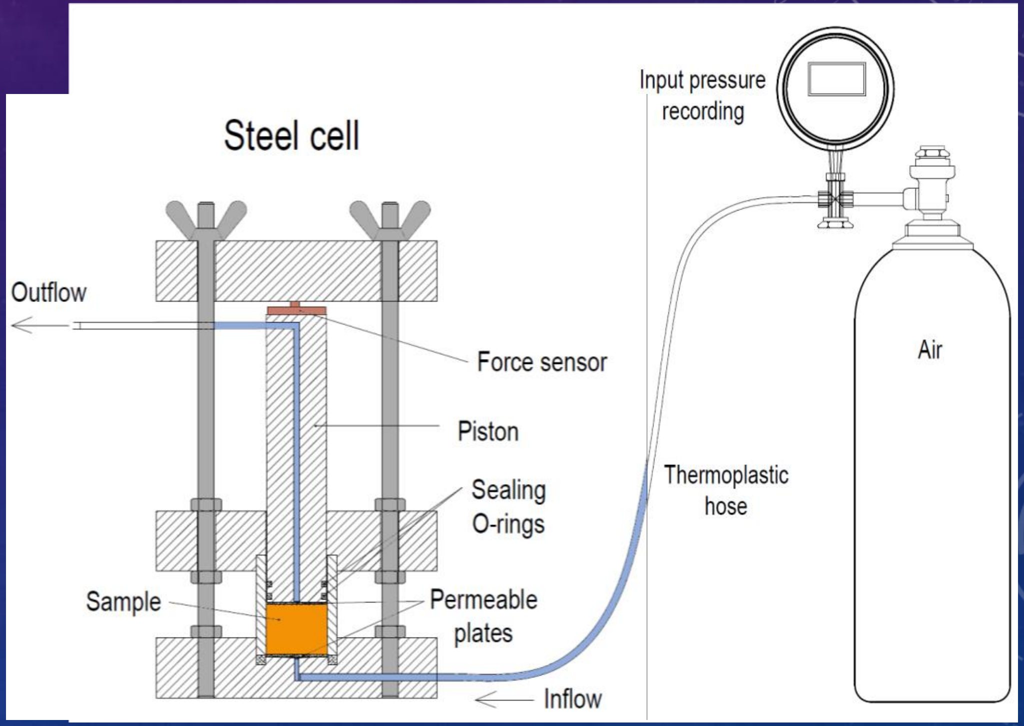
T3.1 - GAS-INDUCED IMPACTS ON BARRIER INTEGRITY

T3.2 - PATHWAY CLOSURE AND SEALING PROCESSES

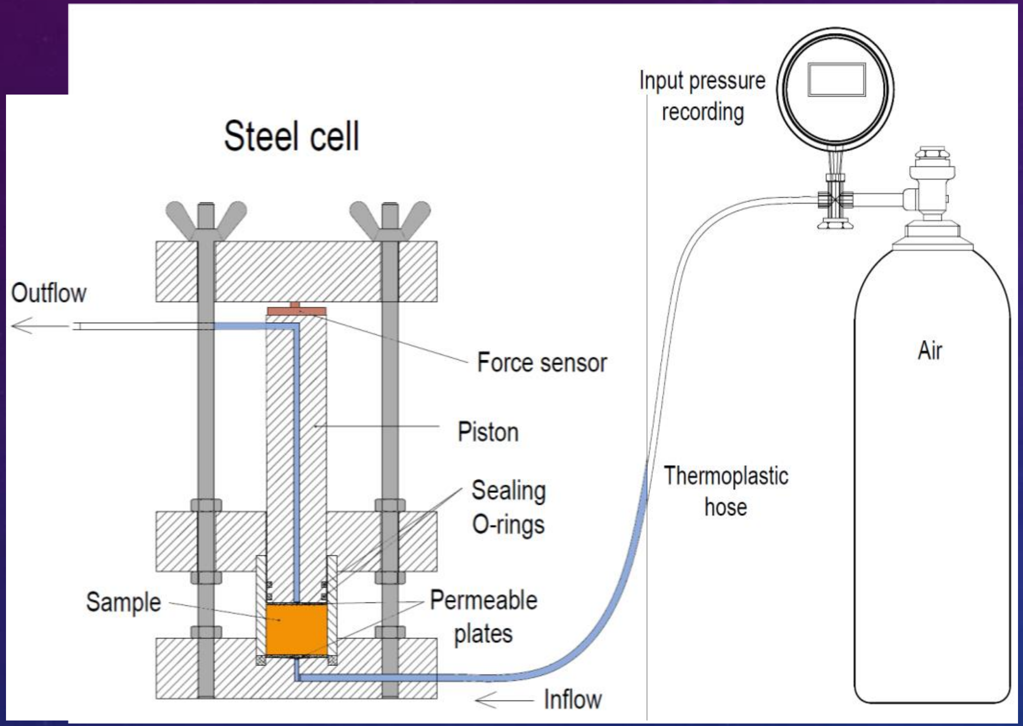
FAST TESTS

How it works?

- **Initial saturation (hydraulic conductivity, swelling pressure)**
- **Gas breakthrough test**
  Monitoring: input gas pressure, total pressure, flow rate at output
- **Re-saturation (hydraulic conductivity, swelling pressure)**
- ……(5 repeated cycles)
- **Dismantling**



FAST TESTS



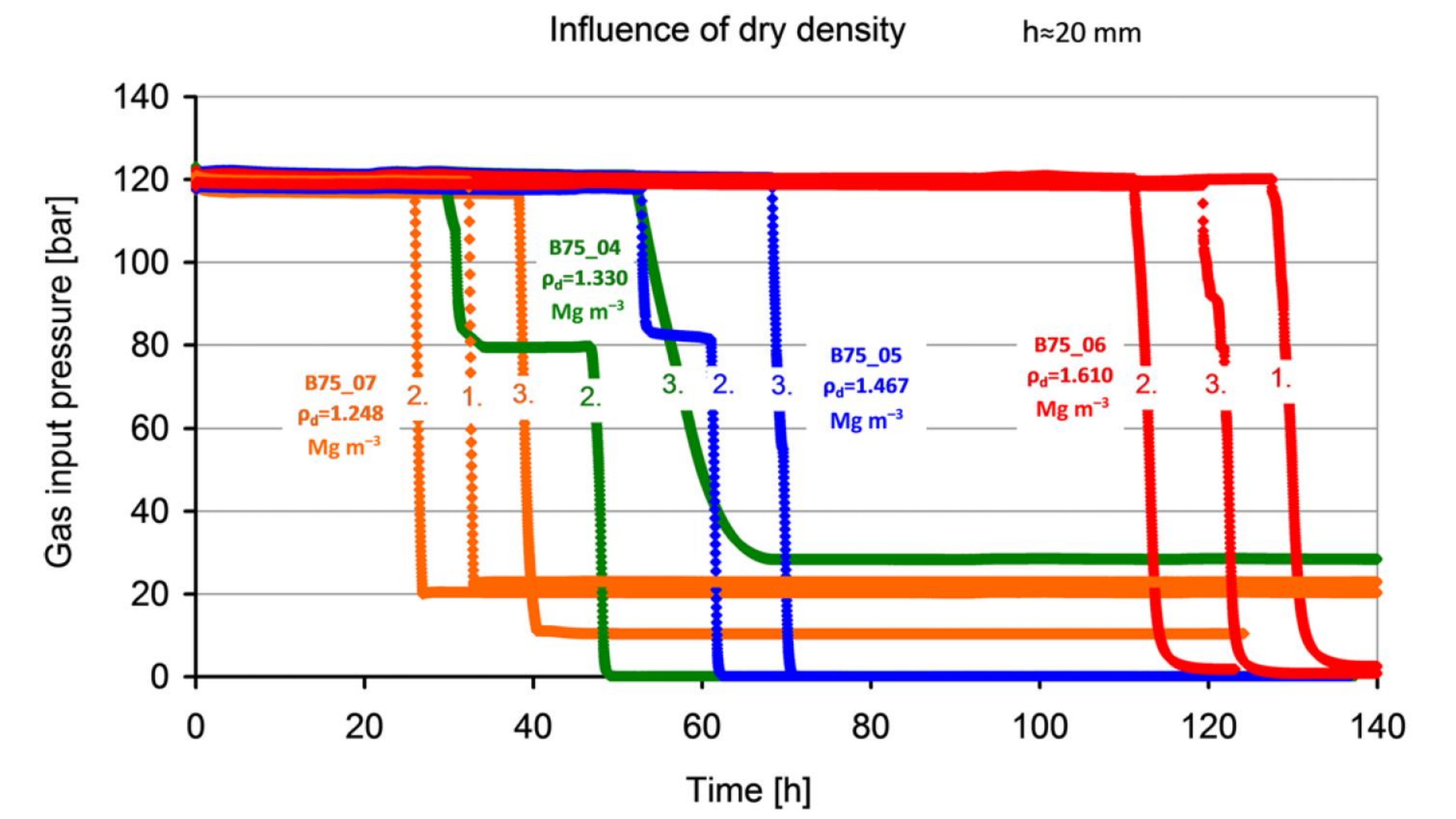
Evaluation

Comparison (between cycles of one sample and between samples) of:

- Time to breakthrough
- Evolution of outflow rate after breakthrough
- Input pressure decay curve after breakthrough (the input line is kept open after breakthrough)
- **Swelling pressure and hydraulic conductivity**

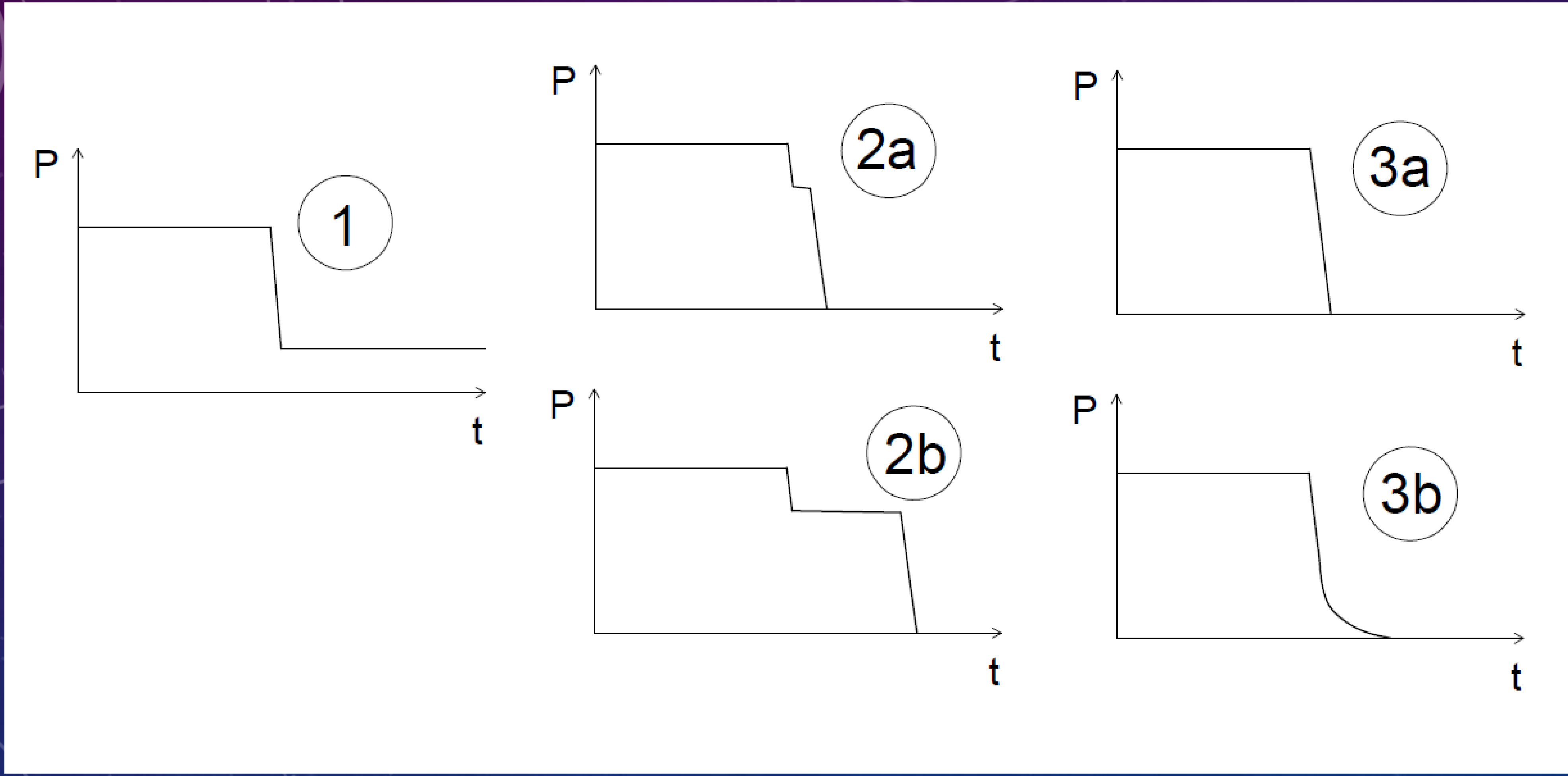
COMPARISON OF REPEATED CYCLES

Bentonite B75 (Czech Ca-Mg bentonite) – project for the Czech Science Foundation (2015)



Smutek, J., Hausmannová, L., Svoboda, J. The gas permeability, breakthrough behaviour and re-sealing ability of Czech Ca–Mg bentonite. Geological Society, London, Special Publications. 2017, 443(1), 333-348. ISSN 0305-8719. DOI:10.1144/SP443.5.

INPUT PRESSURE DECAY CURVE



COMPARISON OF REPEATED CYCLES

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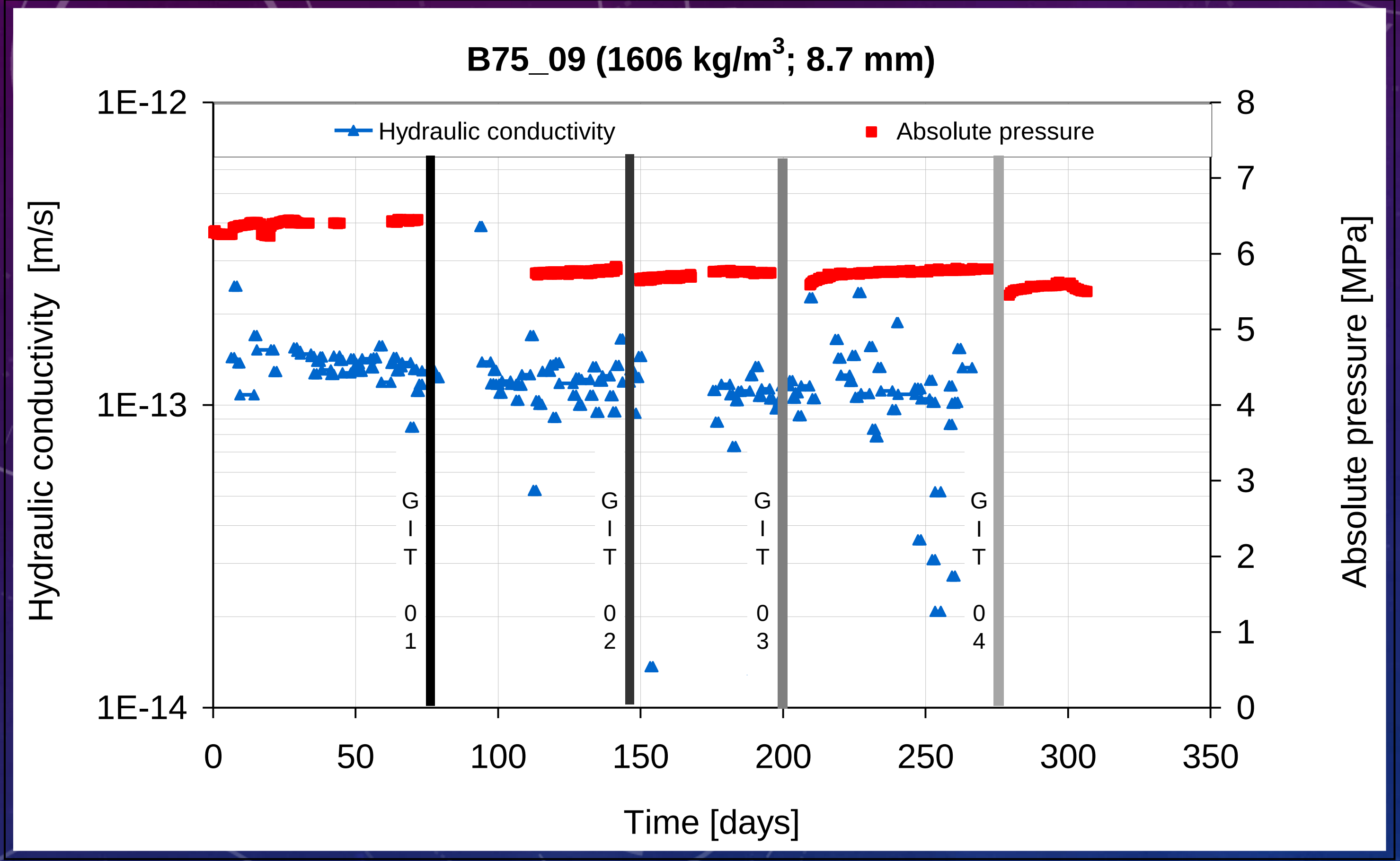

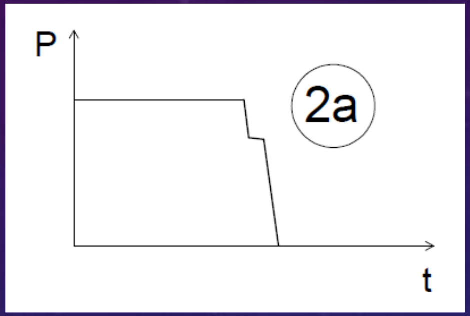

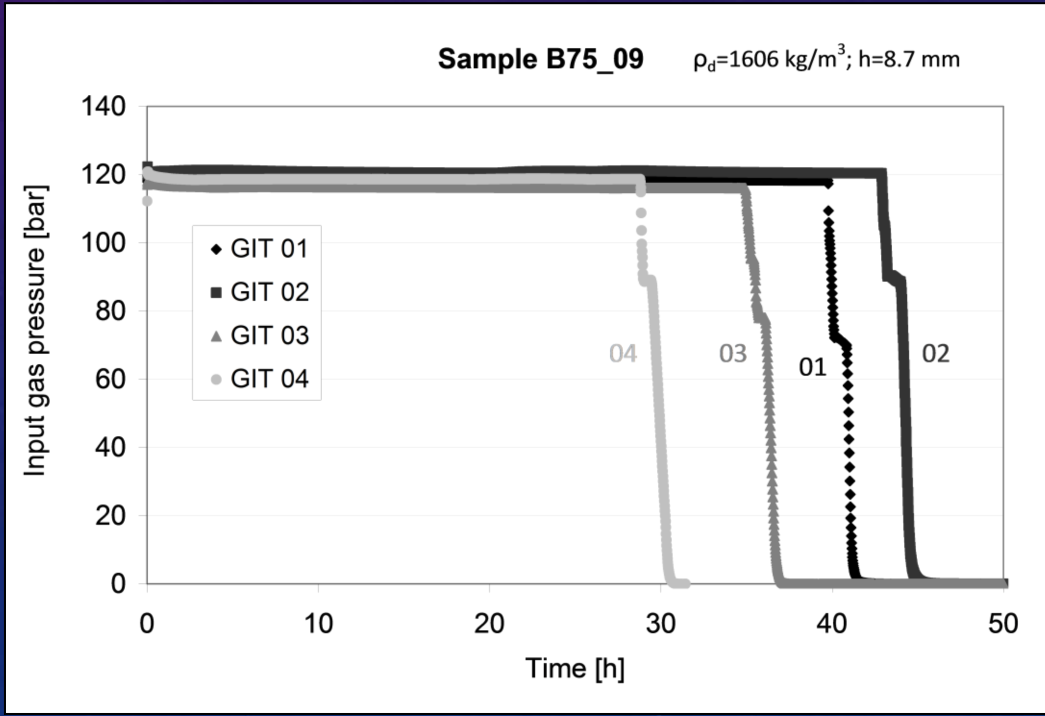

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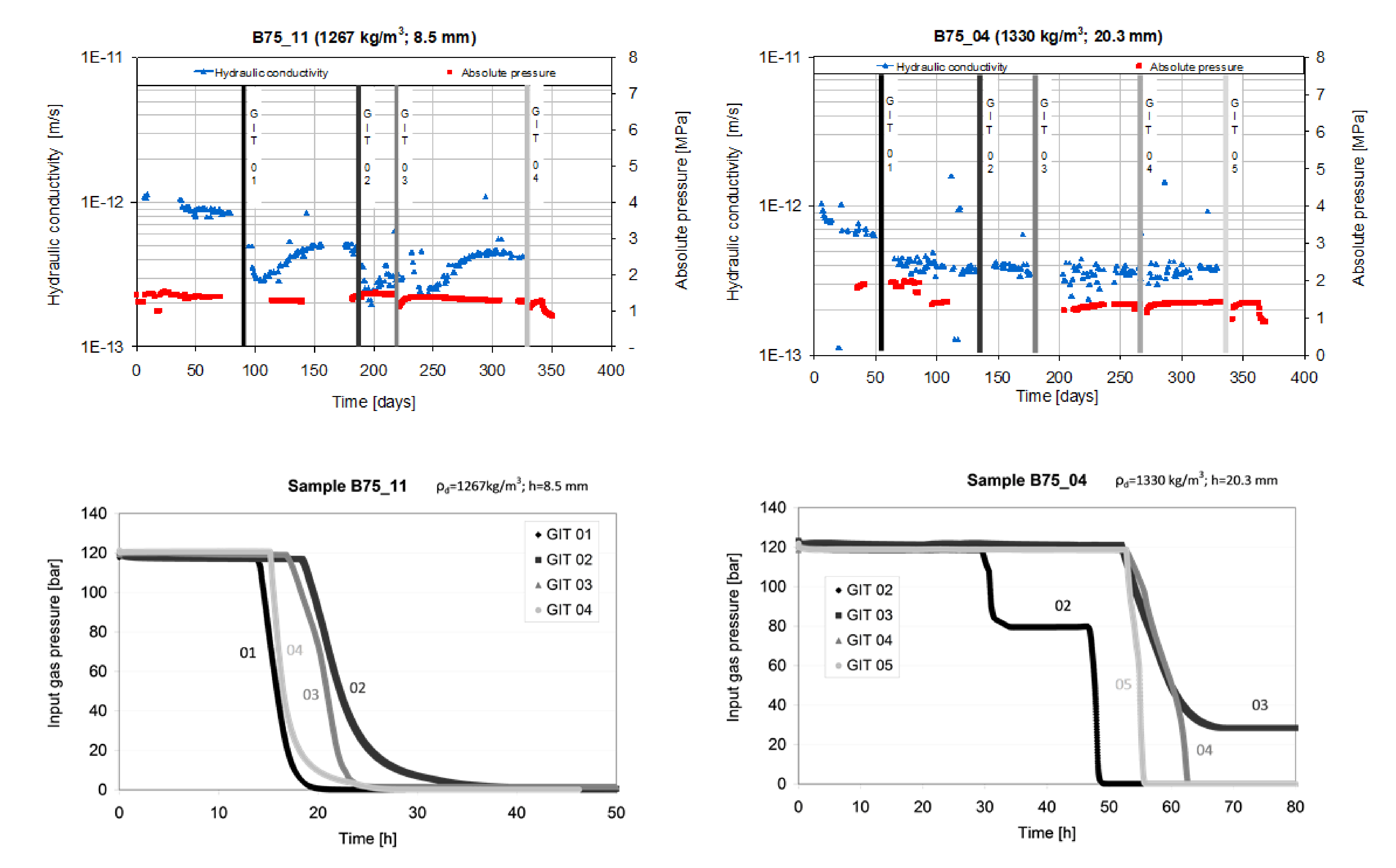
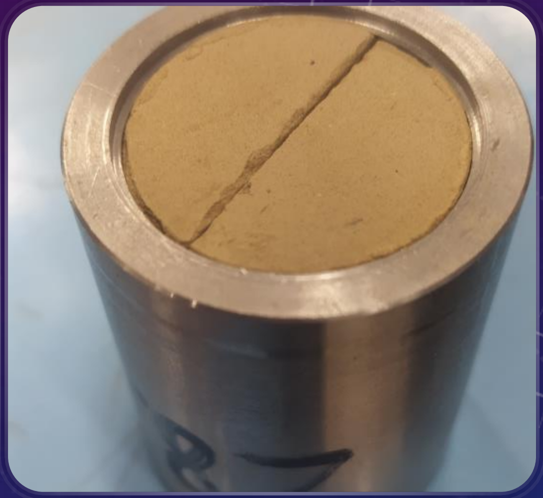


WP GAS

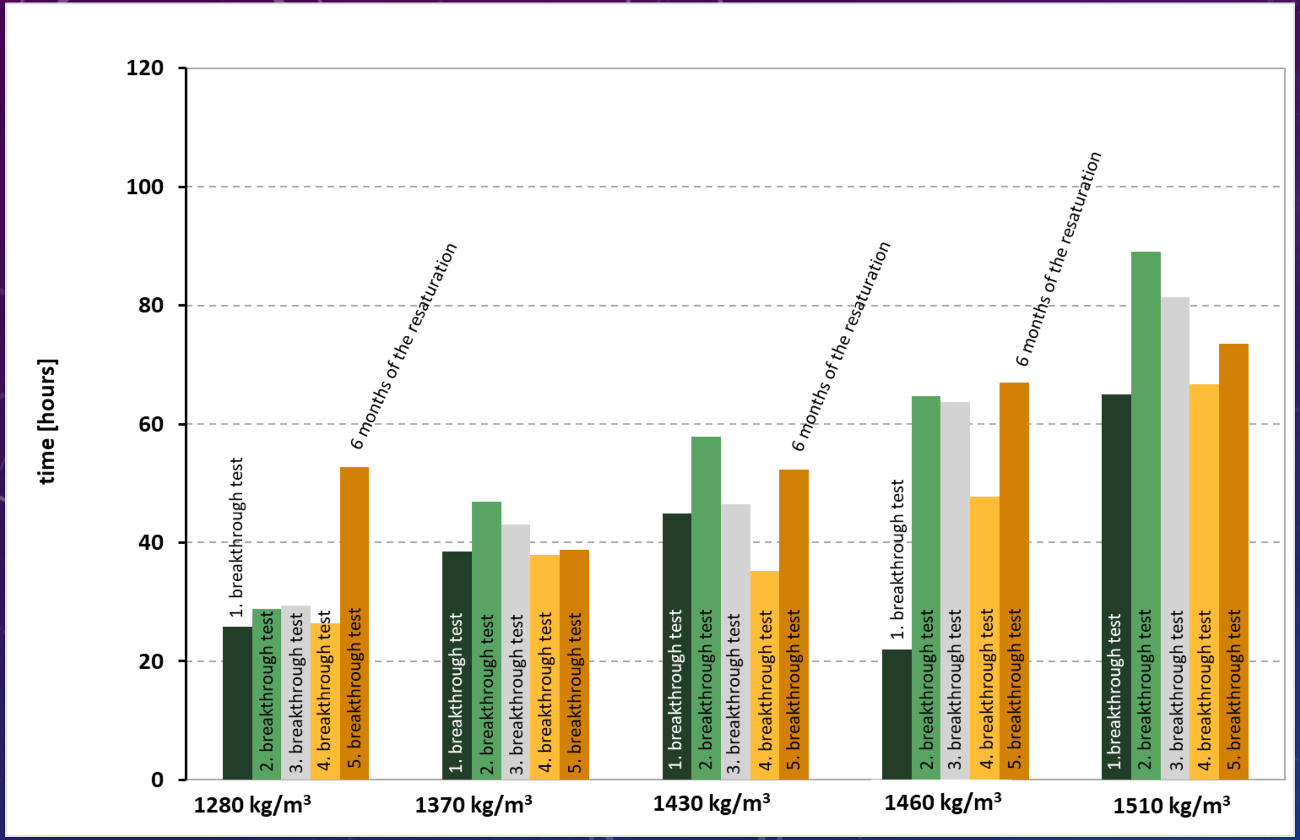
T3.1 - Gas-induced impacts on barrier integrity
T3.2 - Pathway closure and sealing processes

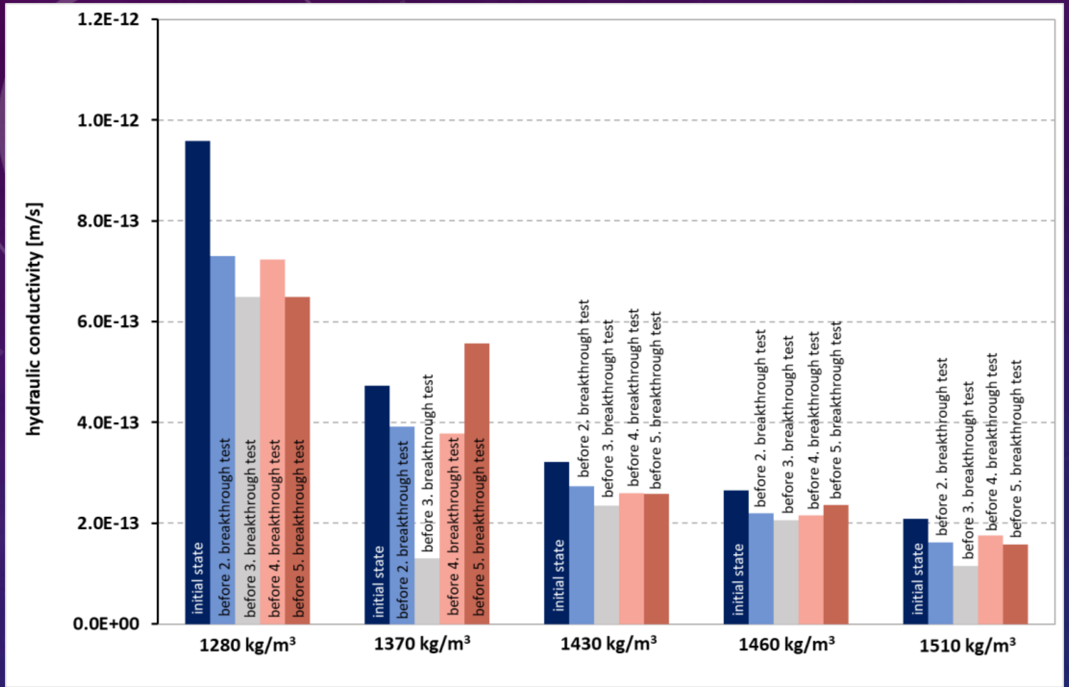
- Homogeneous compacted bentonite samples
  - BCV bentonite
  - 5 samples: dry density 1300 - 1610 kg/m^3
  - Completed (5 cycles of gas injection and resaturation)

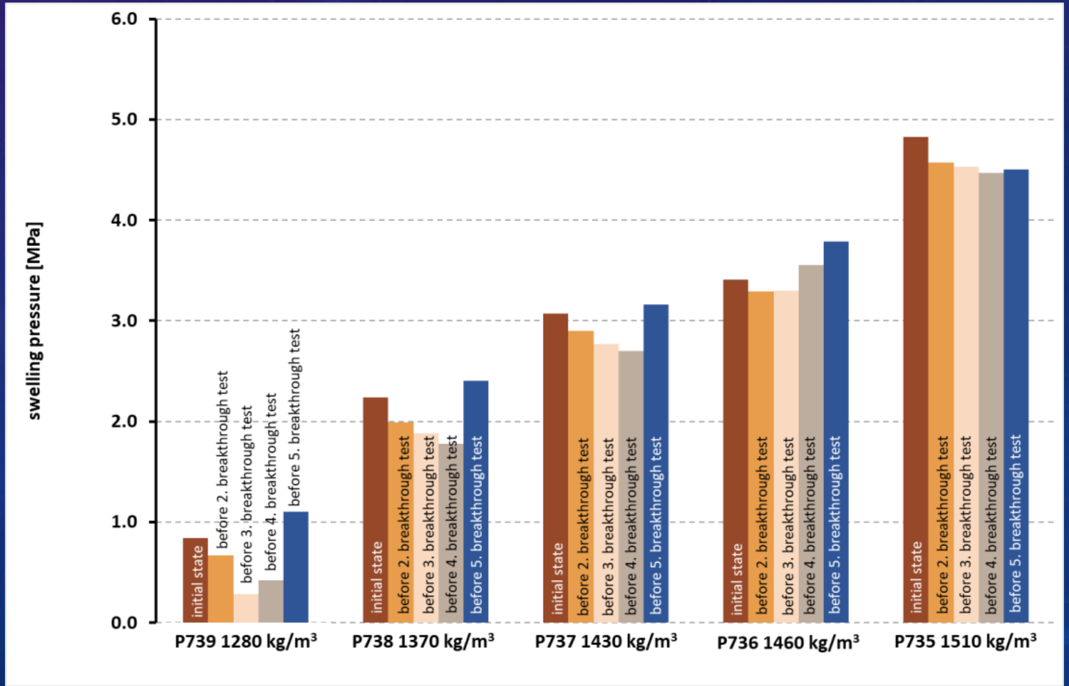


- Inhomogeneous samples (artificial joint)
  - BCV bentonite
  - 4 samples: dry density 1450 - 1610 kg/m^3
  - Ongoing, max. 3 cycles finished
  - The next breakthrough test series planned to June 2023

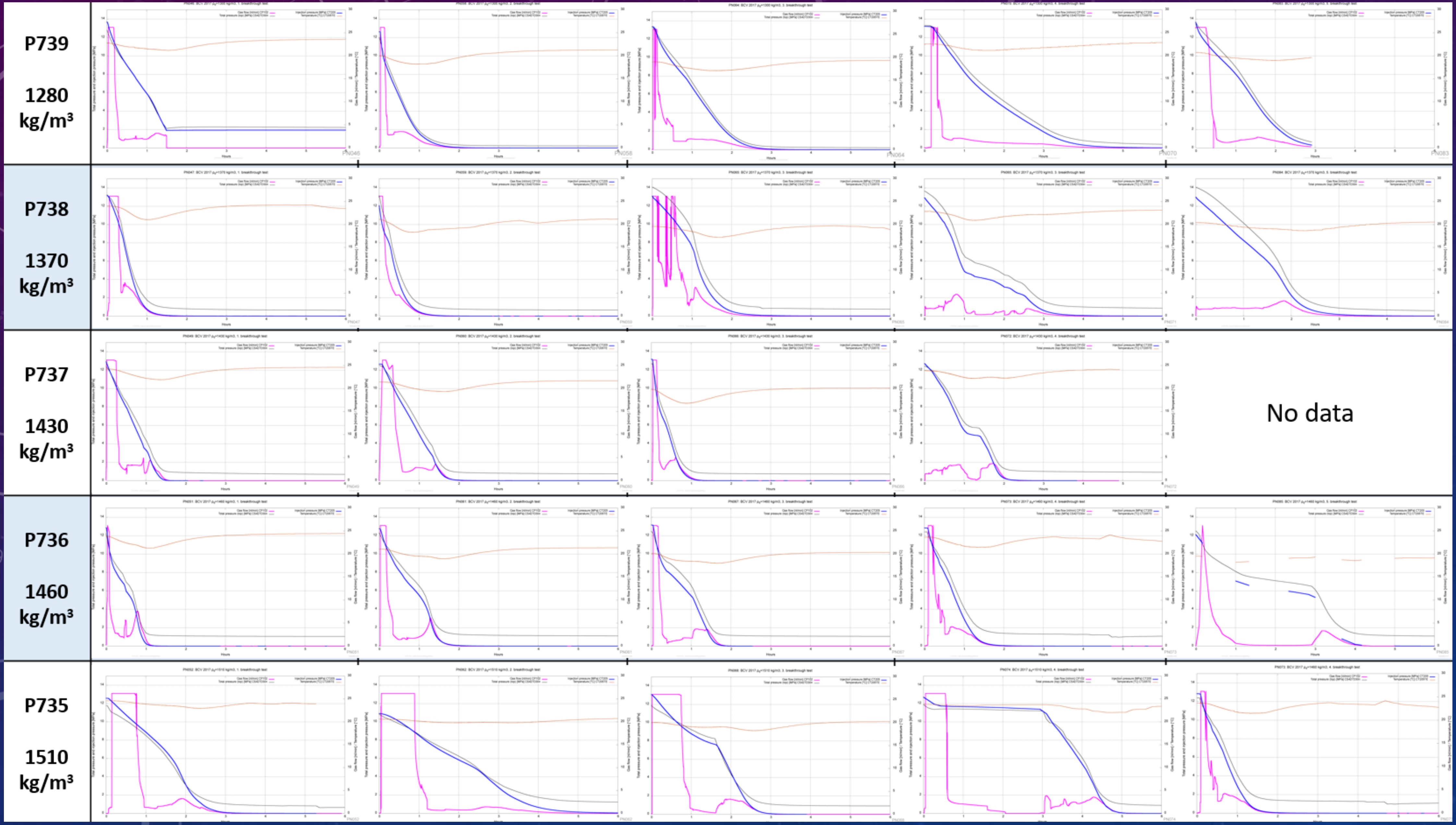
HOMOGENEOUS SAMPLES



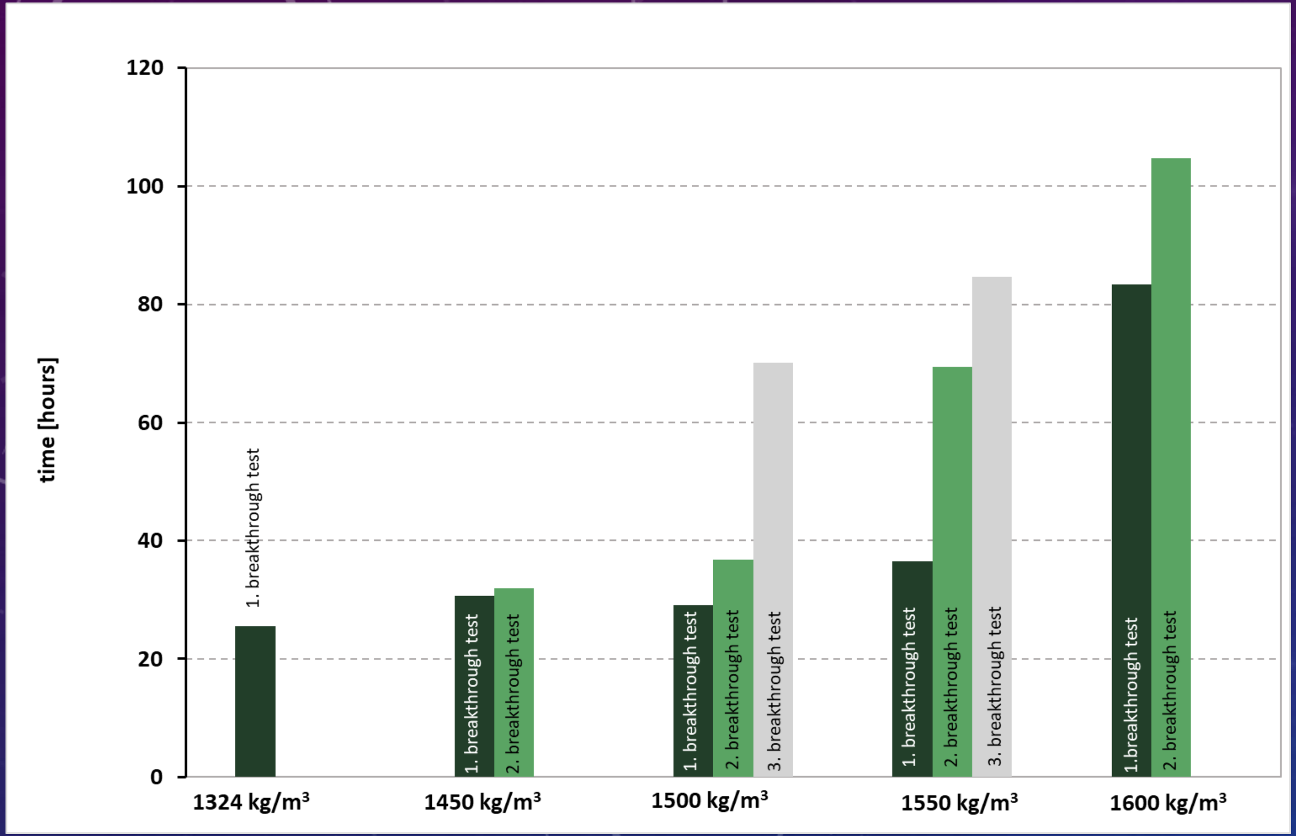


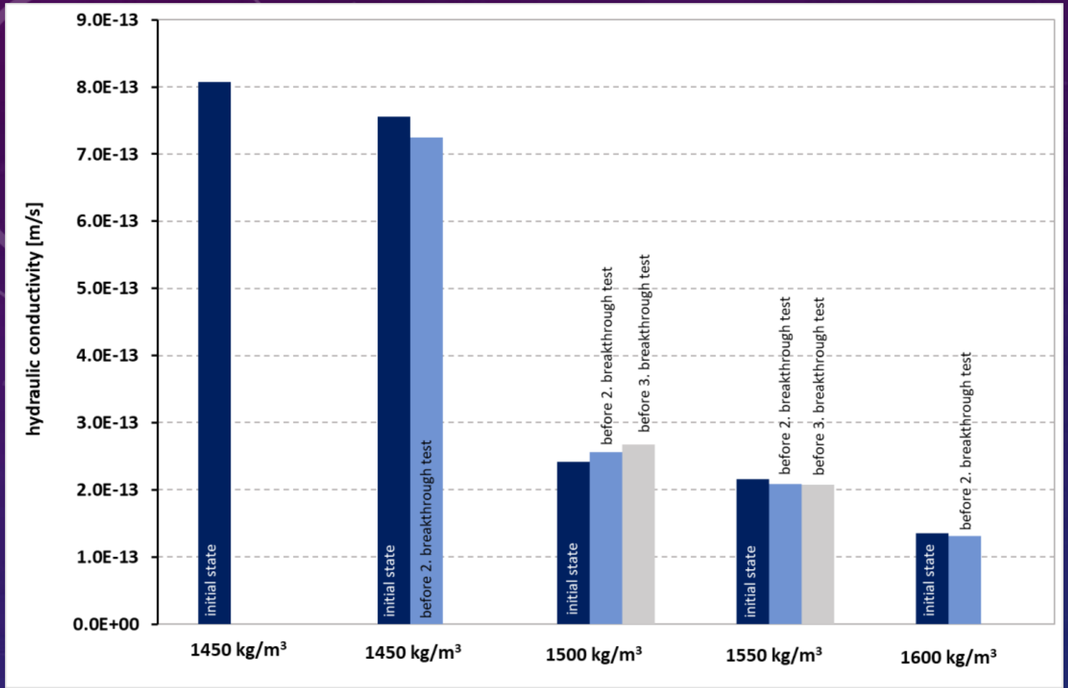


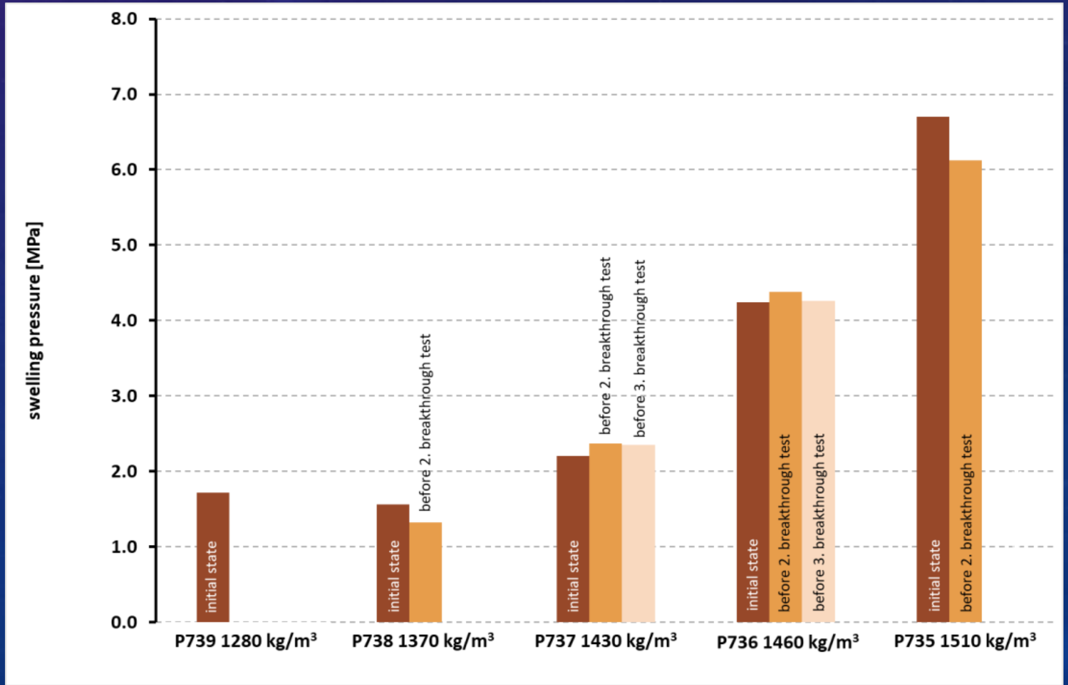
HOMOGENEOUS SAMPLES – BREAKTHROUGH EPISODES



INHOMOGENEOUS SAMPLES







CONCLUSIONS

- Integrity of barrier seems to hold after gas breakthrough given enough time (resaturation)
- Duration of saturation has an impact on the self-healing of the sample
- Fast test can be used to check EBS state and resilience. The endurance in the fast test is a qualitative indication of the EBS state
- Gas tests show clearly that bentonite evolves long time even whet hydraulic conductivity and pressure is stable

LET'S CONTINUE WITH HITEC - TEMPERATURE

The overall objective is to evaluate whether an increase of temperature is feasible and safe by applying (i) existing and (ii) the within the task newly produced knowledge about the behaviour of clay buffer materials at elevated temperatures.

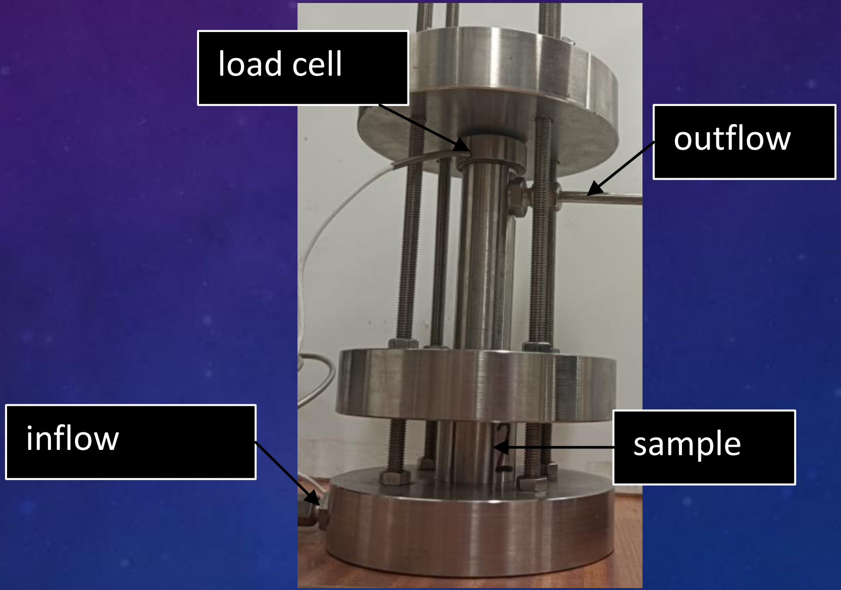
The increase of temperature may result in strong evaporation near the heater and vapour movement towards the external part of the buffer. As a consequence, part of the barrier, or all of it, depending on the particular disposal concept, will remain unsaturated and under high temperatures during periods of time that can be very long. Moreover the high temperature gradient (and pore pressure) even crossing boiling point of water will lead to several adverse effects as Sauna effects.

The aim is to gain knowledge to hydro-mechanical behaviour at high temperature. The temperature impact on important processes will be measured either while the clay is at the high temperature or after a high temperature exposure. Processes that may have a temperature dependence are swelling pressure, hydraulic conductivity, erosion properties, transport of solutes etc.

- T3.1 Characterization of material treated by high temperature
- T3.2 Determination of parameters at temperatures >100°C
- T3.3 Small scale experiments, model development and verification

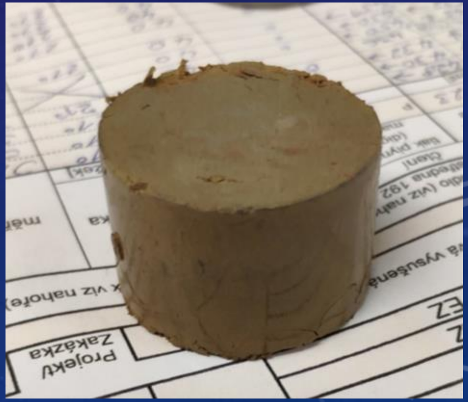
HITEC – T3.1 MATERIAL TREATED BY HIGH TEMPERATURE

- Swelling
  - Free swelling
  - Swelling pressure
- Hydraulic conductivity
- Atterberg limits
- Composition



CTU cell

The sample after the test,
d = 30 mm, h = 20 mm



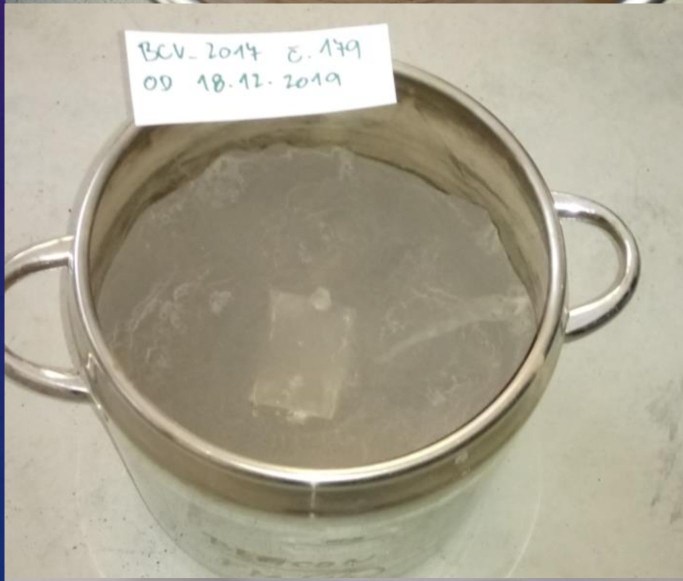
BCV MATERIAL

HITEC – influence of high temperature

- Dry material @150°C
- Suspension @150°C

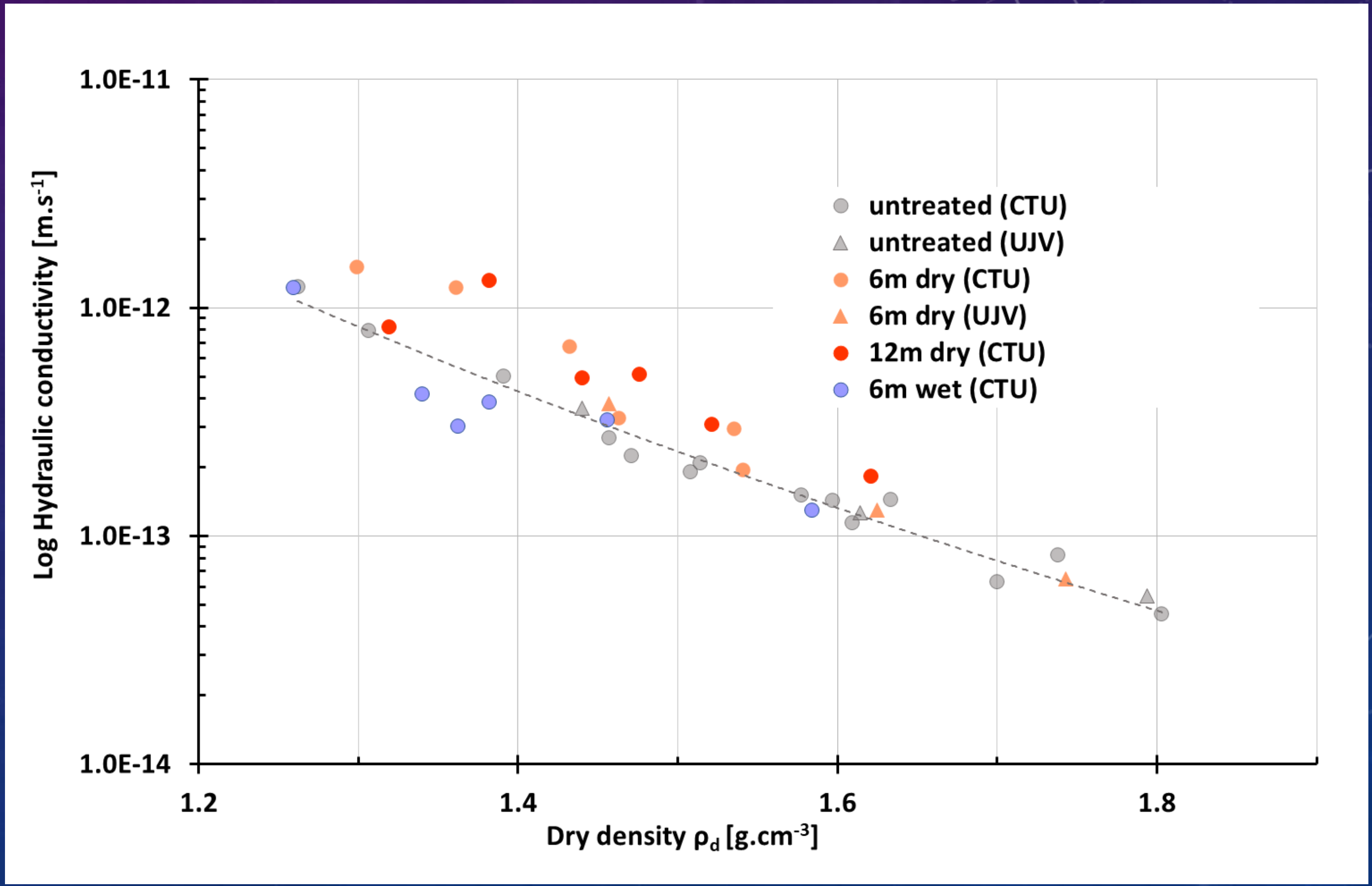
Sampling:

- 6 months
- 12 months
- 24 months



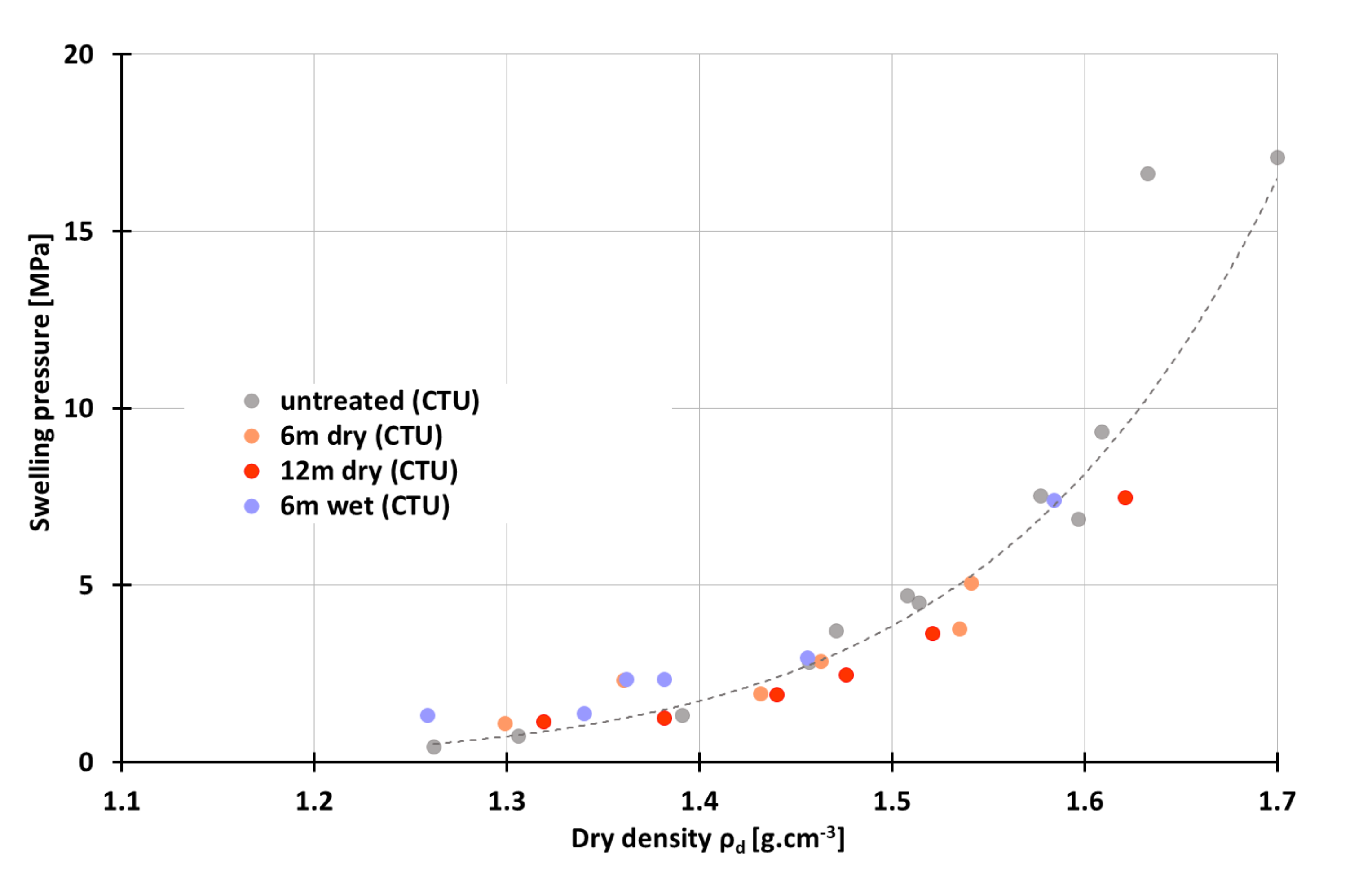
HYDRAULIC CONDUCTIVITY

- Hydraulic conductivity of thermally treated BCV in dry state is systematically above the trend line of untreated BCV
- No difference between *k* of dry treated BCV after 6m of treatment and *k* after 12m of treatment is observed
- No impact of elevated temperature on wet treated BCV is observed. In part of low densities the measured values are under the trend line of untreated BVC



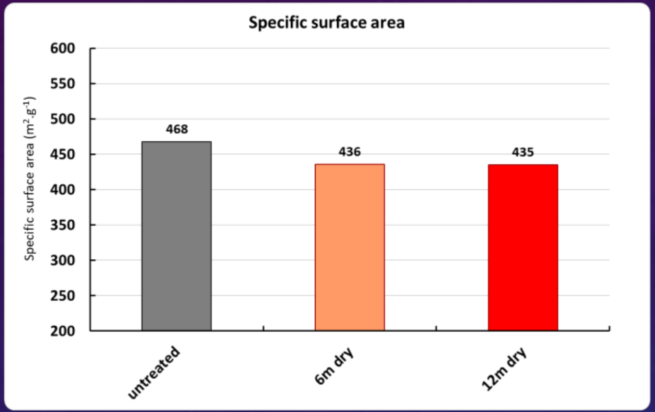
SWELLING PRESSURE

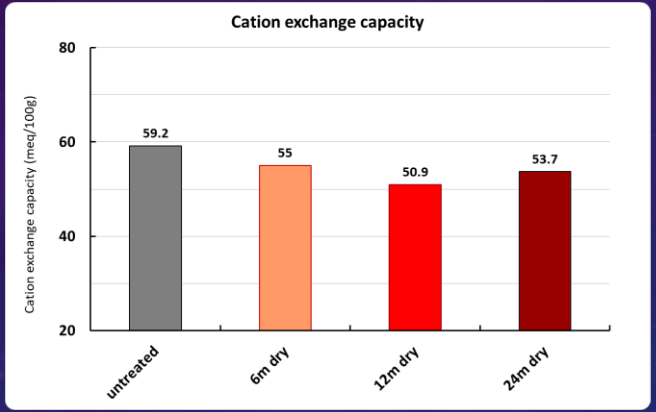
- Consistent decrease of swelling pressure is observed on set of samples of dry treated bentonite
- No impact of duration of thermal treatment is observed on dry treated bentonite
- No significant difference in swelling pressure is observed between wet treated and untreated bentonite

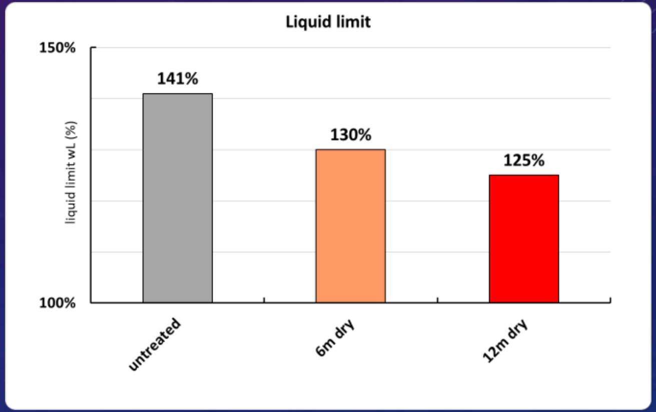


WL, CEC, SSA AFTER THERMAL TREATMENT @150 °C

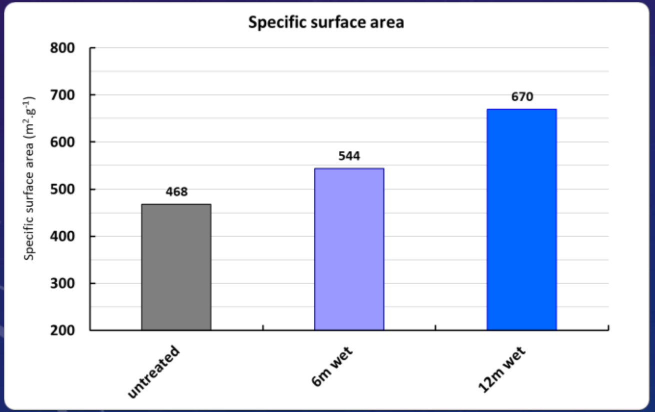
- Same trend observed for liquid limit (cone method), cation exchange capacity (Cu-trien method), specific surface area (EGME) and hydraulic conductivity

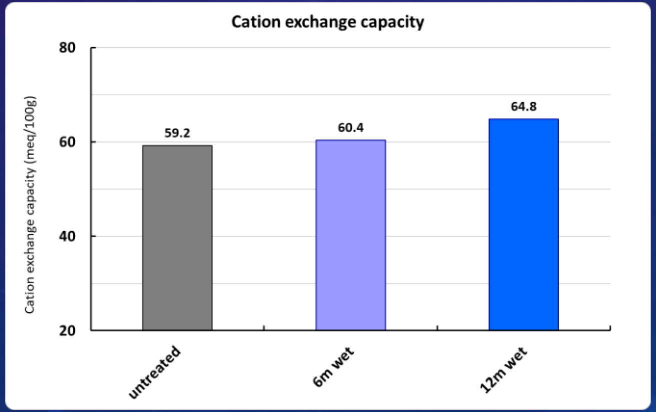


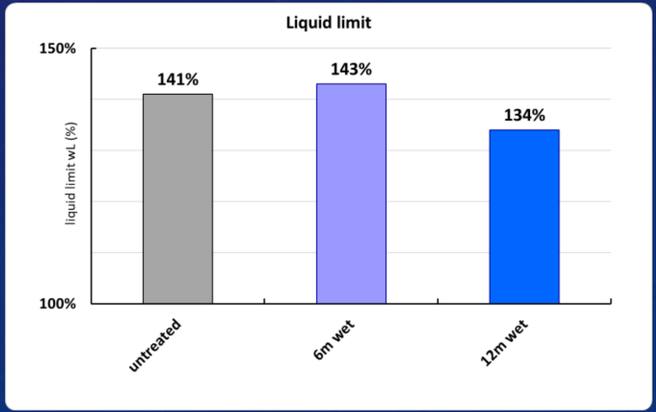




DRY treated





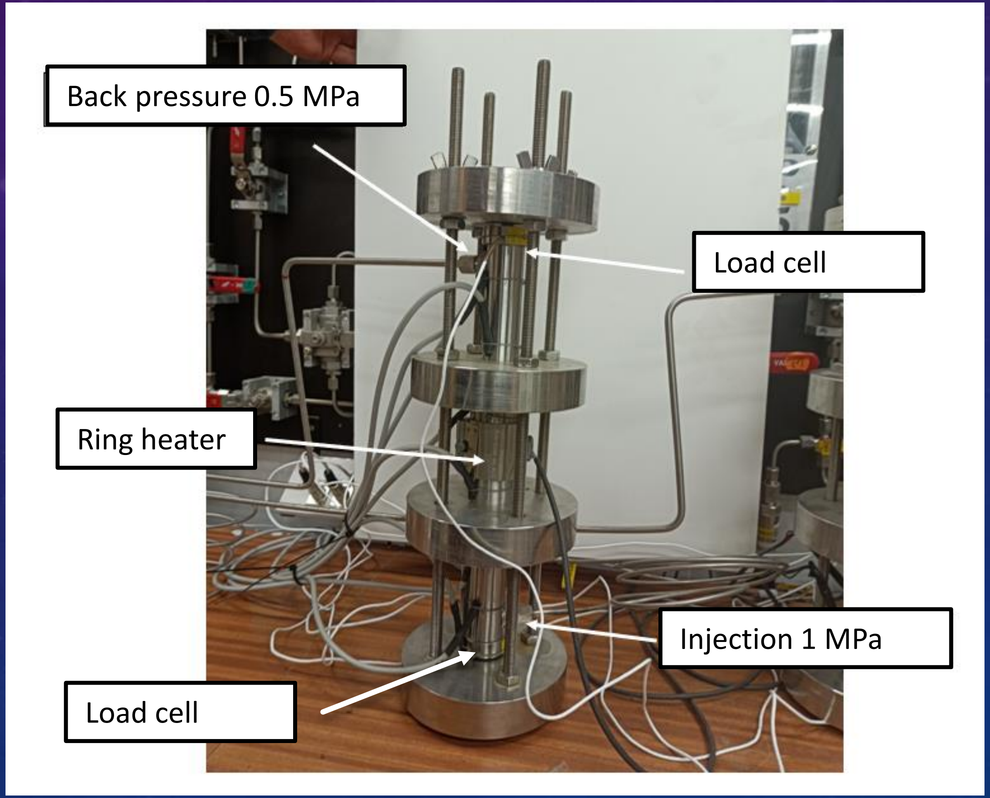


WET treated

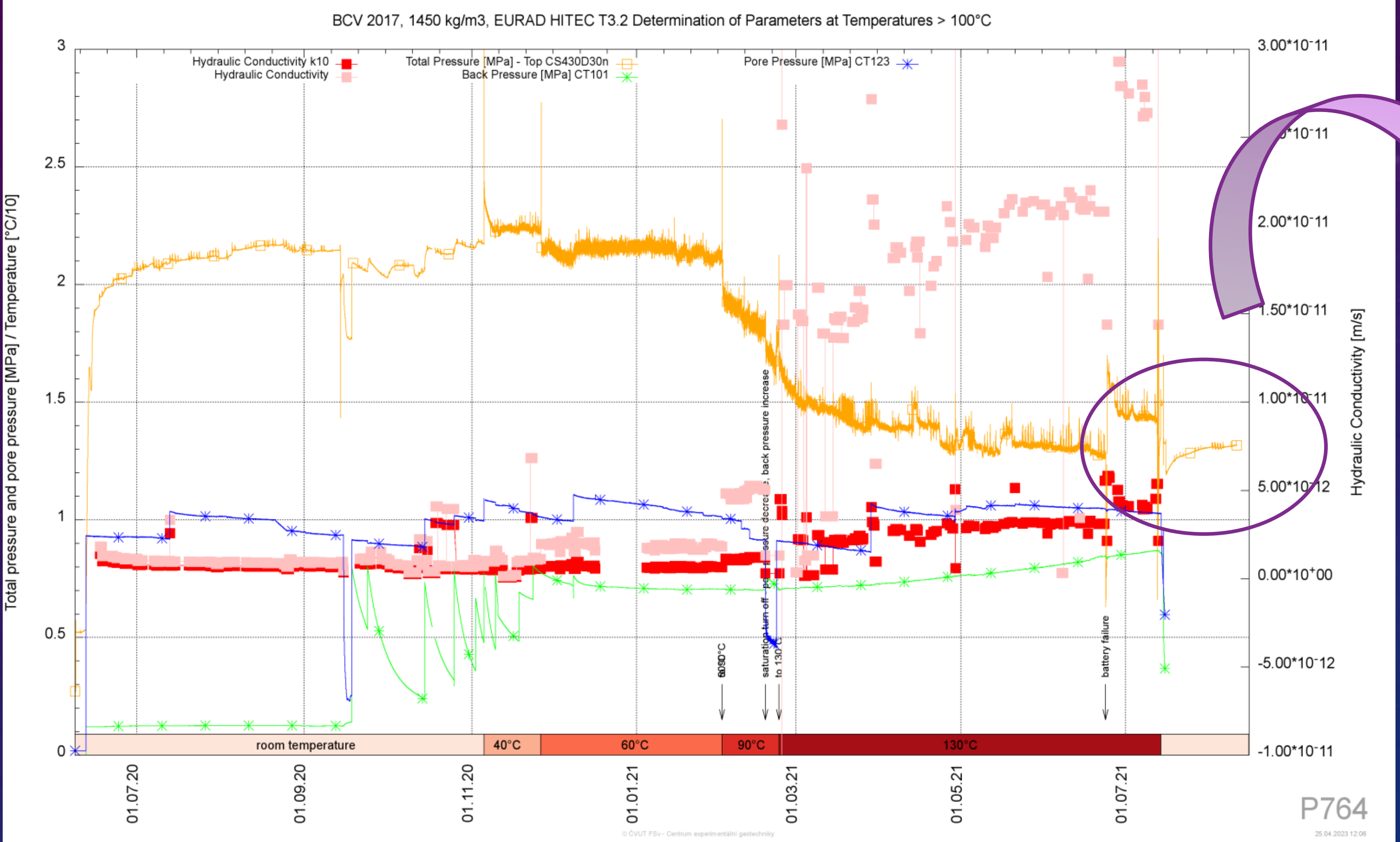
HITEC – T3.2 DETERMINATION OF PARAMETERS AT TEMPERATURES >100°C

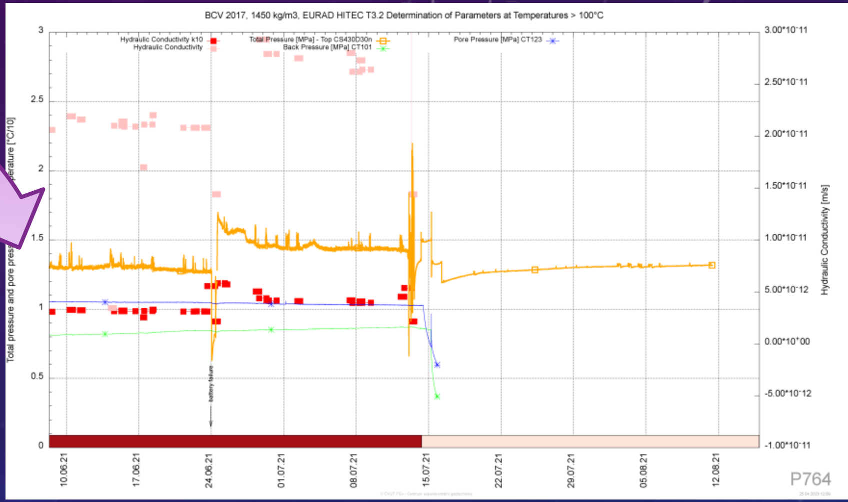
- Swelling pressure
- Hydraulic conductivity

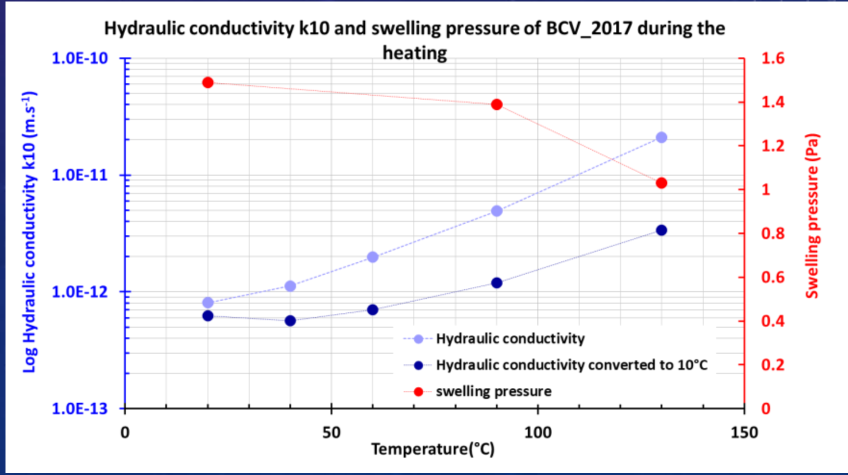
- Temperature up to 130°C
- Start at laboratory temperature



BCV @130 °C

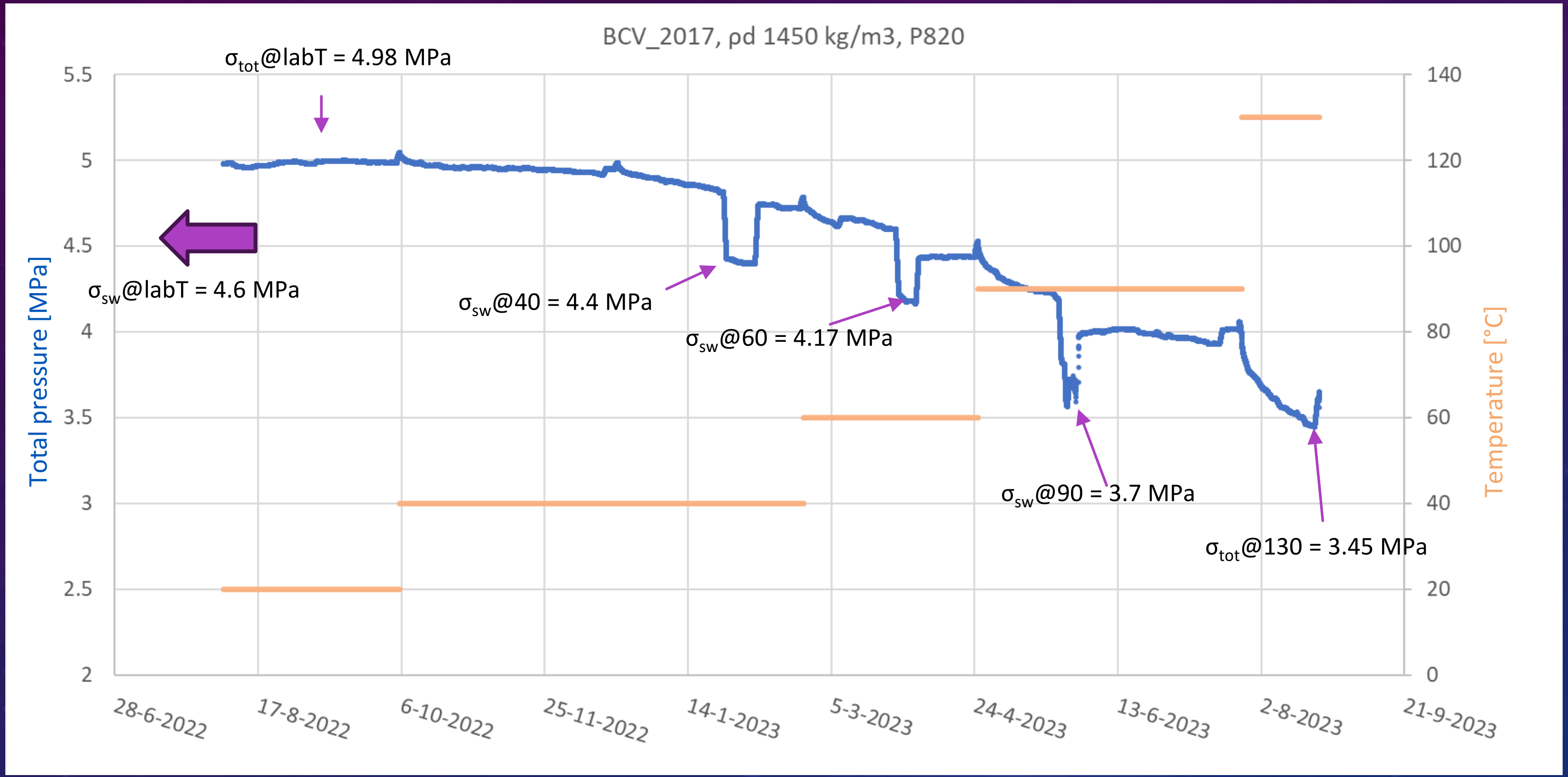


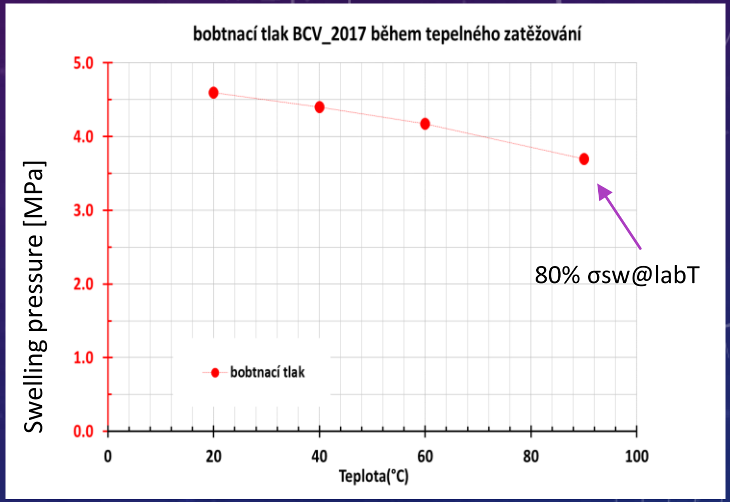




- Continuous decrease of total pressure
- Swelling pressure does not recover to the values of untreated material

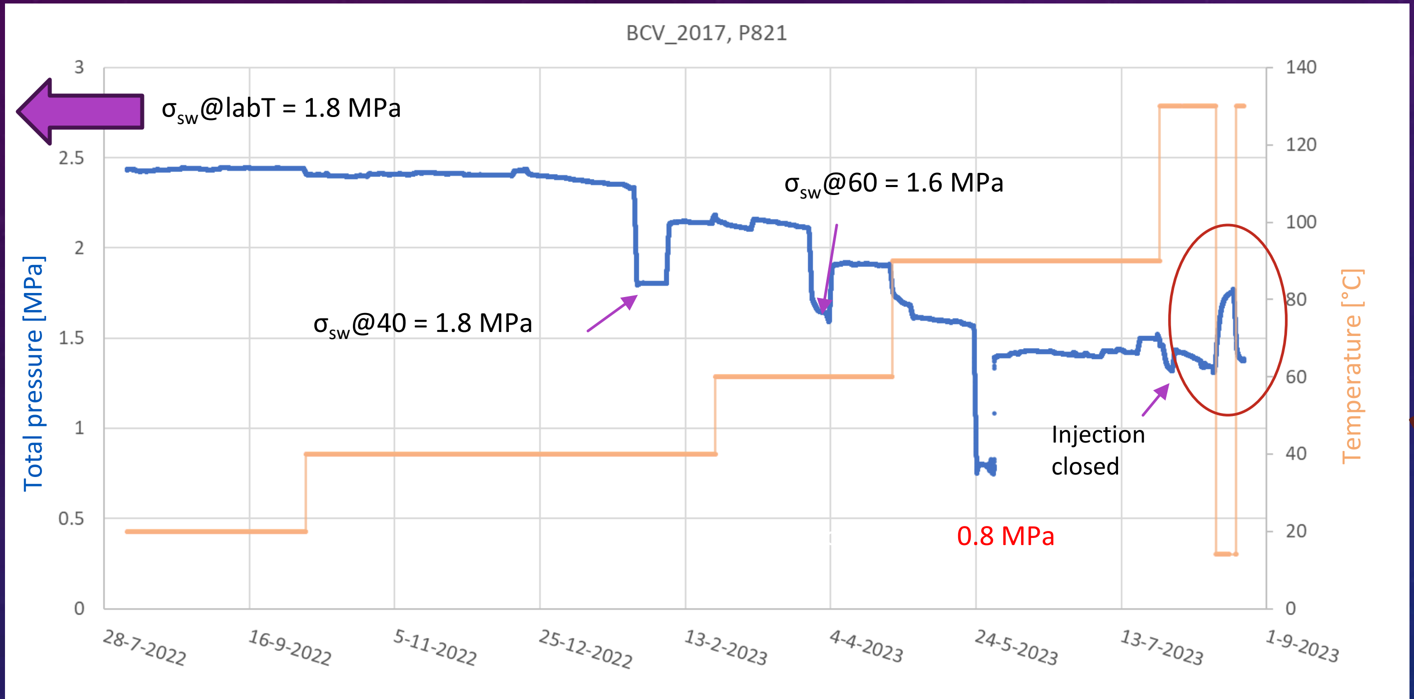
BCV @130 °C

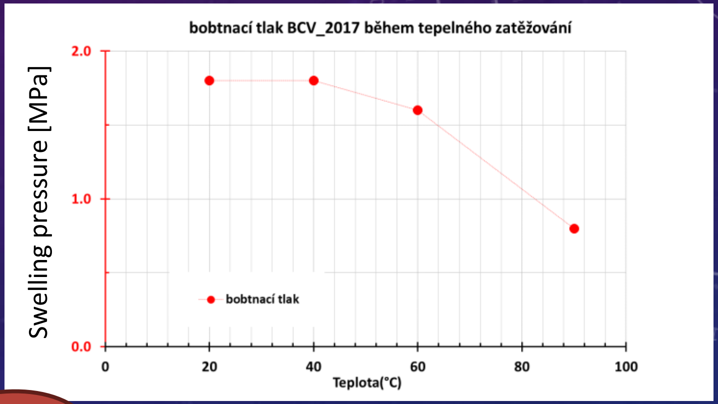




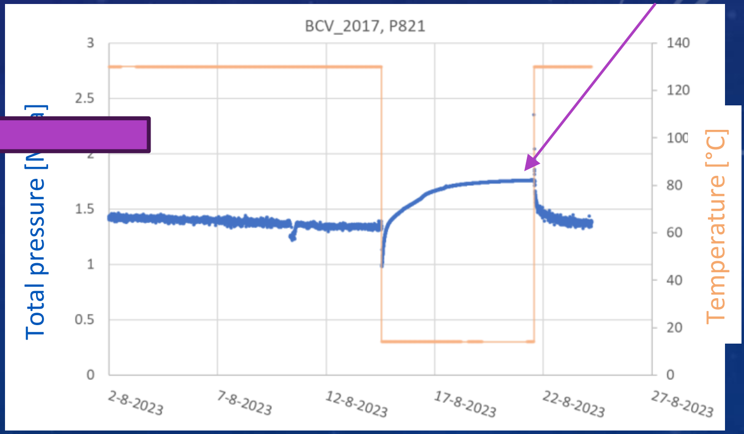
- Continuous decrease of total pressure

BCV @130 °C





σ_{tot}@labT after themal load = 1.76 MPa



σ_{tot}@labT before thermal load = 2.43 MPa

T	σ_{sw} (MPa)	%σ_{sw}@labT
lab	1.8	100
40 °C	1.8	100
60 °C	1.6	89
90 °C	0.8	44

- Continual decrease of total pressure

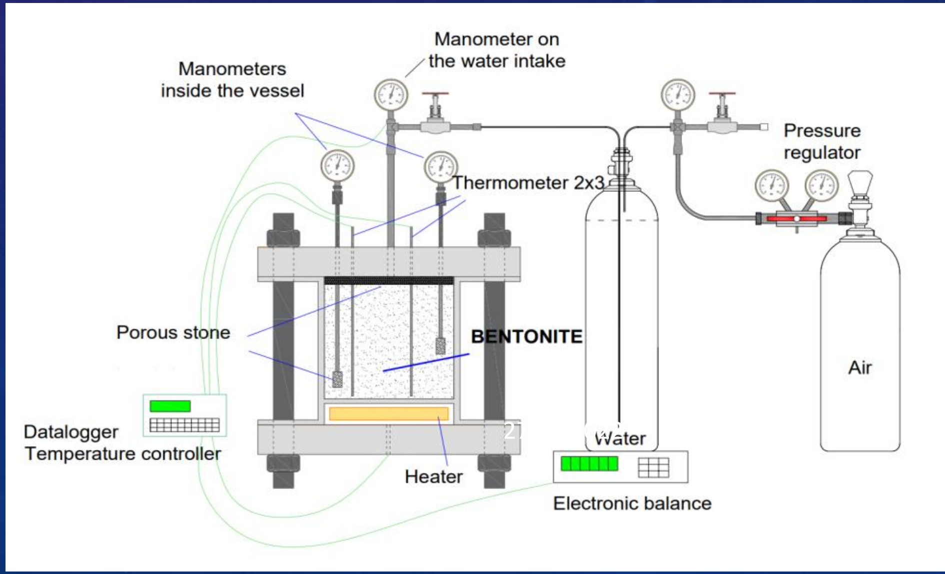
T3.3 SMALL SCALE EXPERIMENTS, MODEL DEVELOPMENT AND VERIFICATION

First run

- Powdered BCV, 900 kg/m^3
- 1. Phase – saturation by 6 bar
- 2. Phase – gradual heating up to 150 °C
- Heating and the saturation at the same time
- No boiling

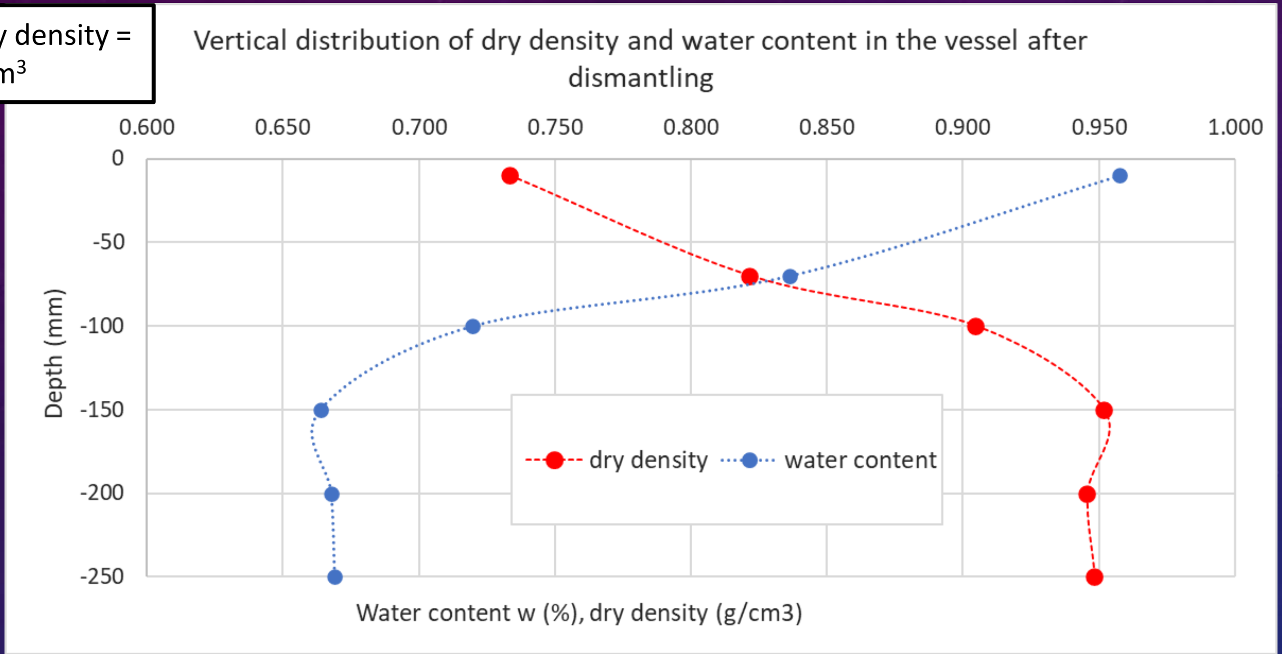
Second run

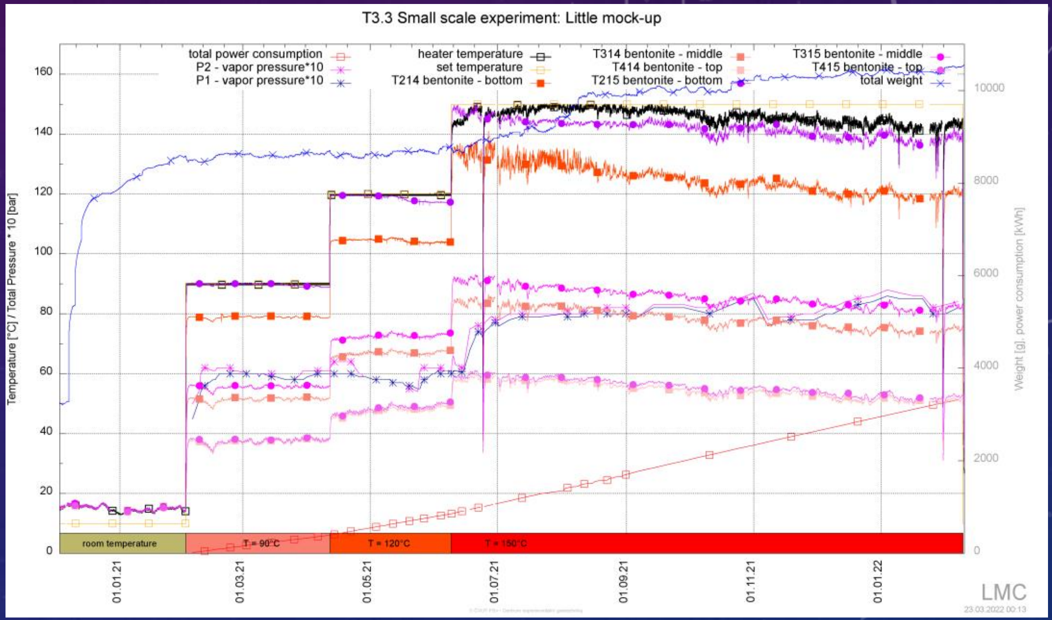
- Peletized BCV, 1400 kg/m3
- 1. Phase – heating right up to 150 °C – simulation of the condition of the repository
- 2. Phase – start of saturation by the pressure ensuring boiling in the middle of the vessel
- Heating and the saturation at the same time
- Boiling



FIRST RUN

Initial dry density = 0,84 g/cm³



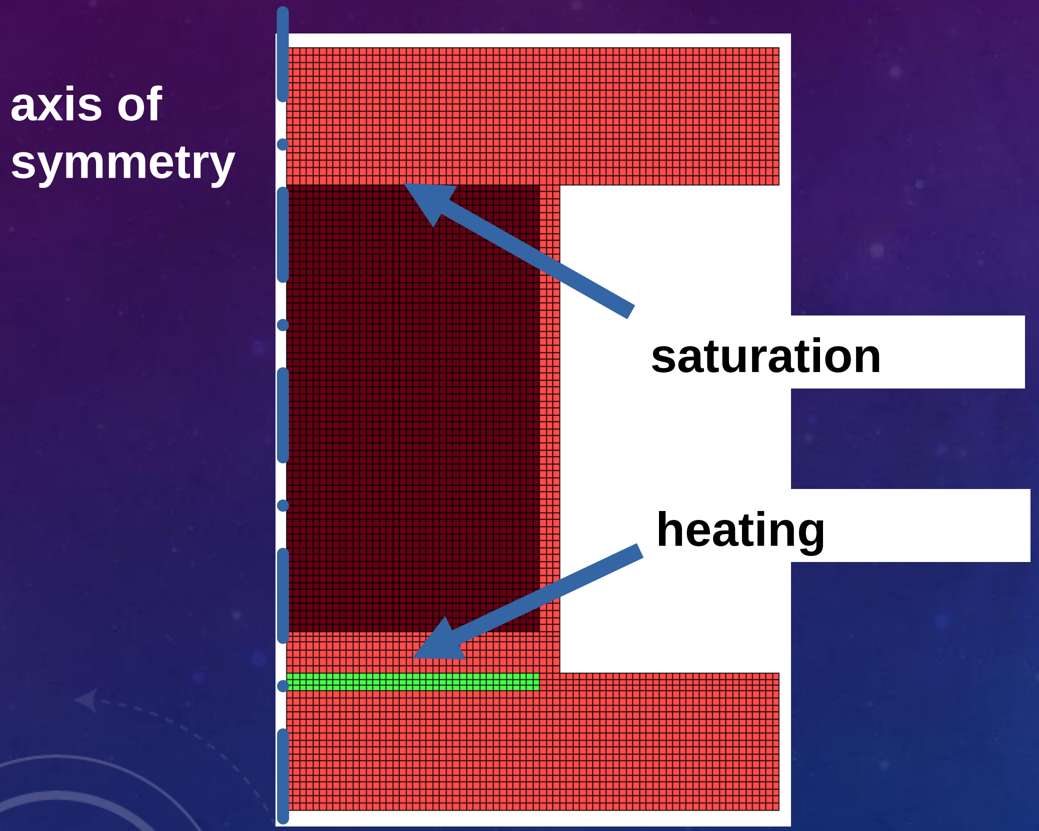


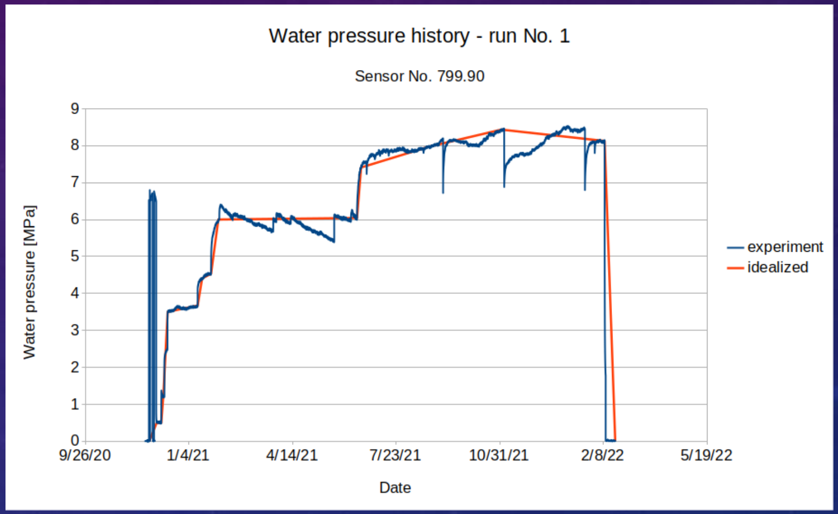
depth (mm)	w (%)	ρ (g/cm3)	ρd (g/cm3)
0			
-10	98%	1.388	0.733
-70	84%	1.507	0.821
-100	72%	1.559	0.904
-150	66%	1.584	0.951
-200	67%	1.568	0.945
-250	67%	1.578	0.948

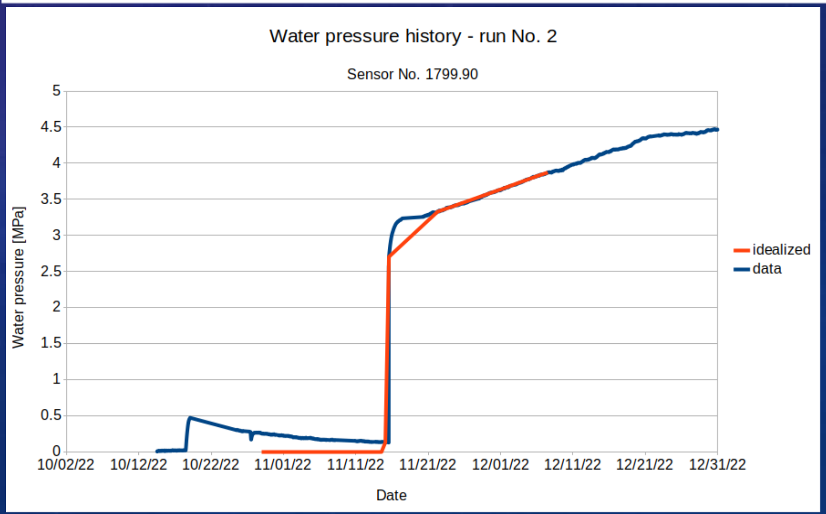
3nd layer (-100 mm)

MAIN RESULTS – NUMERICAL MODELLING

- FE model – axisymmetric domain with the same geometry for the first and the second runs





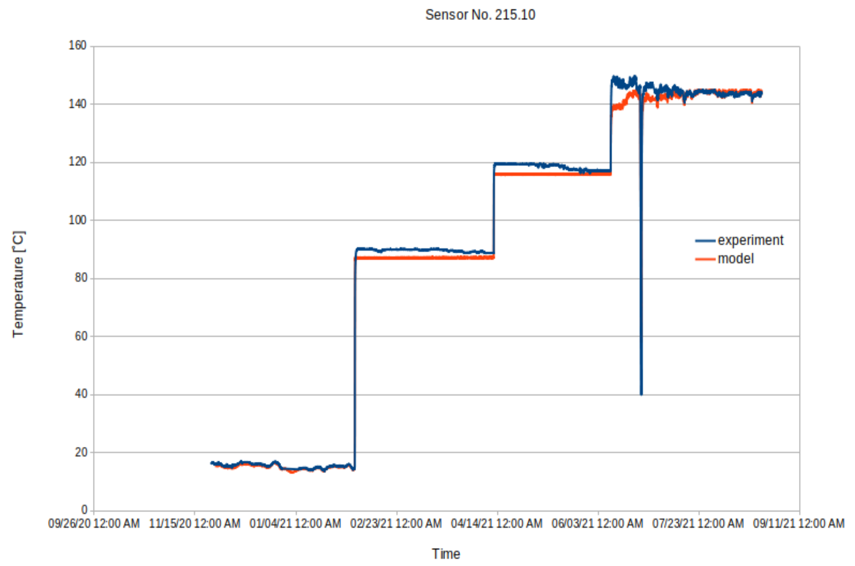


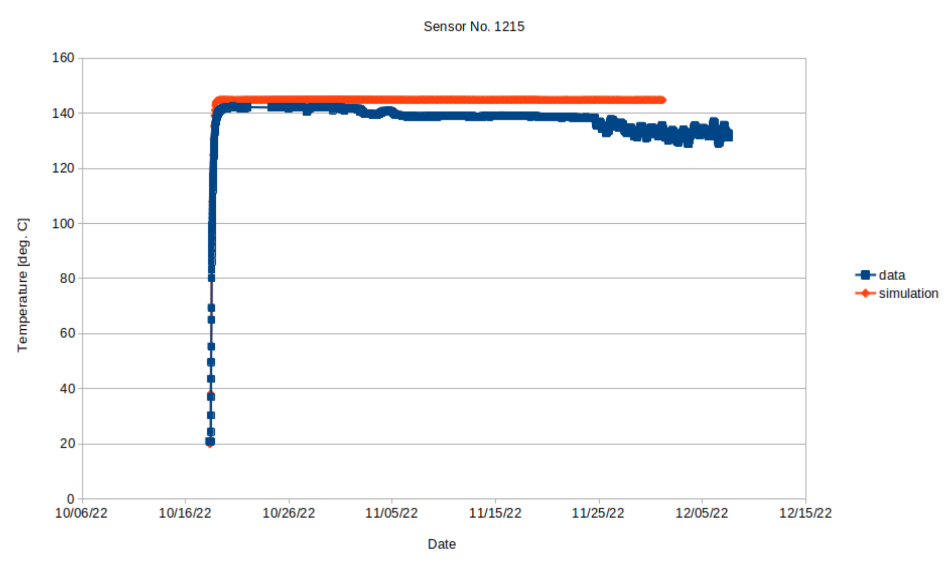
NUMERICAL VS REAL

First run

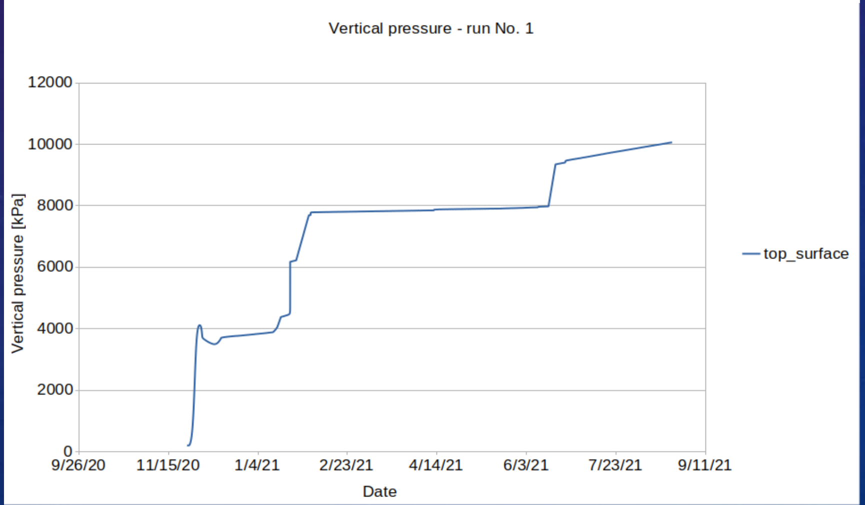
Second run

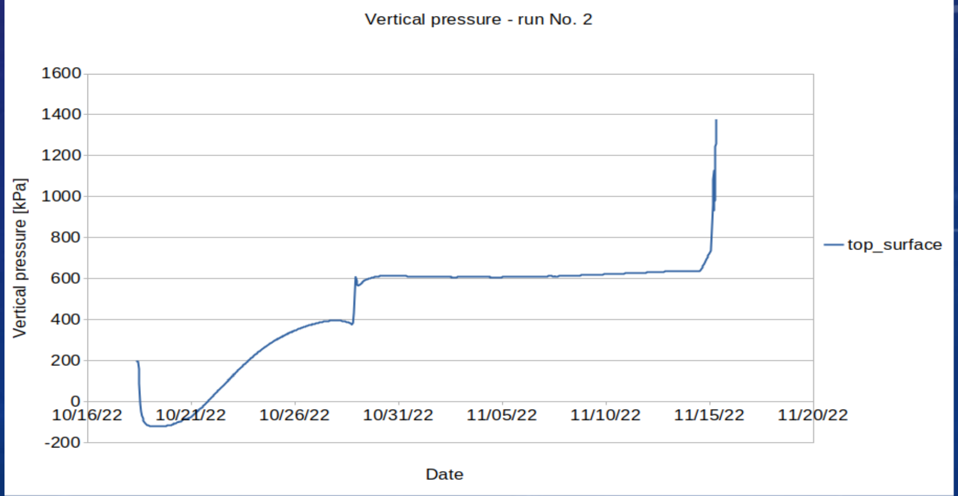
- Temperature





- Pressure





COMBINATION OF TEMPERATURE AND GAS?

Under investigation…

- First results show that fast tests have lower time to breakthrough. Probably coinciding with observed decrease of swelling pressure.

THANK YOU FOR YOUR ATTENTION

END OF PRESENTATION

ACKNOWLEDGEMENT

This project has received funding from the European Union´s Horizon 2020 research and Innovation programme 2014-2018 under grant agreement N°847593

BCV testing was also supported by the Euratom research and training programme 2014-2018 under contract no. 745942 Bentonite Mechanical Evolution

And more testing of BCV was supported by the project Engineered barrier 200C (no. TK01030031) from the Technology Agency of the Czech Republic

B75 testing was supported by Czech Science Foundation (project 14-19655S)