Methodology and technical design of the Bukov URF corrosion experiment

Dobrev et al.

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SUPPLIERS:

ÚJV Řež, a. s.[1], Vysoká škola chemicko-technologická v Praze (University of Chemistry and Technology, Prague)[2], Technická univerzita v Liberci (Technical University of Liberec)[3], Česká geologická služba (Czech Geological Survey)[4]

AUTHORS: Dobrev D.[1], Hofmanová E.[1], Mendoza Miranda A. N.[1], Jankovský F.[1], Kašpar V.[1], Kouřil M.[2], Pusztai M.[3], Steinová J.[3], Stoulil J.[2], Šachlová Š.[1], Zelinková T.[4], Zuna M.[1]

Name and Surname

Markéta Dohnálková (SÚRAO)

4 February 2022

Name and Surname

David Dobrev (ÚJV Řež, a. s.)

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SÚRAO

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Abbreviations:

16S rRNA	gene/marker for ribosomal RNA
ASV	amplicon sequence variant
ATP	adenosine triphosphate
BCV	bentonite from the Černý vrch deposit
CEC	cation exchange capacity
DGR	deep geological repository
DNA	deoxyribonucleic acid
EDU	Dukovany nuclear power plant
EGME	ethylene glycol monoethyl ether
Eh	redox potential, related to a reference (hydrogen) electrode
ETE	Temelín nuclear power plant
HMTA	hexamethylenetetramine
ICP-MS	inductively coupled plasma mass spectrometry
IRB	iron-reducing bacteria
NGS	next generation sequencing
NPP	nuclear power plant
NRB	nitrate reducing bacteria
OES	optical emission spectrometry
ORP	oxidation-reduction (redox) potential
OTU	operational taxonomic unit
PCR	polymerase chain reaction
PTFE	polytetrafluoroethylene
qPCR	quantitative PCR
SAED HR-TEM	selected area electron diffraction – high resolution transmission electron microscopy
SEM/EDS	scanning electron microscopy with energy-dispersive X-ray spectroscopy
SI	swell index
SNF	spent nuclear fuel
SRB	sulphate-reducing bacteria
SSA	specific surface area
TA-EGA	thermal analysis with evolved gas analysis
TC	total carbon
TIC	total inorganic carbon
TOC	total organic carbon
TS	total sulphur
URF	underground research facility
WDP	waste disposal package
XRD	X-ray diffraction analysis
XRF	X-ray fluorescence spectroscopy

Abstract

The aim of the report is to provide a methodological description of the preparation and installation of an in-situ corrosion experiment to be performed at the Bukov URF employing the materials anticipated for the construction of the Czech waste disposal package (WDP). The report contains descriptions of the apparatus to be used, the experimental gallery characterisation procedure, the material characterisation procedure, the preparation and installation of the heated corrosion experiment, the planned analysis and the uncertainties inherent in the experiment.

Keywords

Methodology, Bukov URF, in-situ corrosion experiment, corrosion, WDP materials, bentonite

1 Introduction

The Czech waste disposal package (WDP) for spent nuclear fuel concept is based on the use of steel materials with acceptable corrosion rates that will guarantee the required service life. The WDP has been designed to feature an outer casing made of carbon steel with a defined chemical composition and strength parameters and a fine-grained structure (for example, S355J2H + N steel meets these requirements). It is anticipated that the inner casing will be made from 316L stainless steel and that each inner casing will house just one fuel assembly. Thus, for SNF from EDU there will be seven inner casings (Fig. 1), while there will be three inner casings for SNF from ETE (Fig. 2). Alternatively, a variant that assumes one stainless steel inner casing for multiple fuel assemblies is still being considered.

In order to verify the proposed concept, it is necessary both to perform a wide range of experiments on the proposed candidate metals and to validate the properties of the bentonite sealing material. Several of the properties of the proposed materials have already been verified via previous research work, but it is important that the results obtained to date be supplemented by the results of in-situ corrosion tests, which will provide information that cannot be provided by laboratory research alone.

Therefore, as part of the *Pilot corrosion experiment at the Bukov URF* project, a long-term heated corrosion test will be performed on the material designed for the construction of the outer casing of the WDP at the Bukov URF, which will also serve for the comparison of the steel material with Cu-OF copper. This study provides a summary of the methodological procedure that will be used in the project for the performance of corrosion testing.
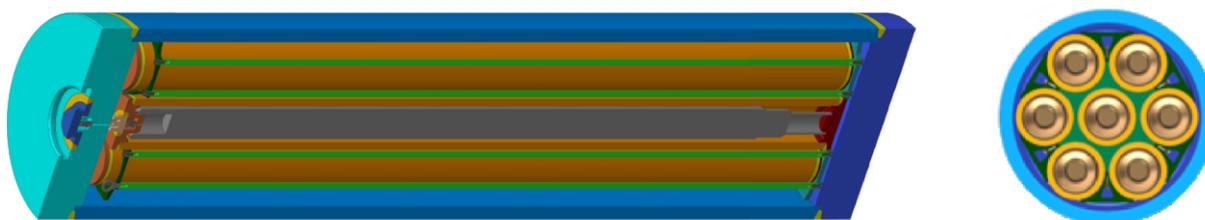
Fig. 1 ŠKODA 440/7 WDP (Forman et al., 2021)
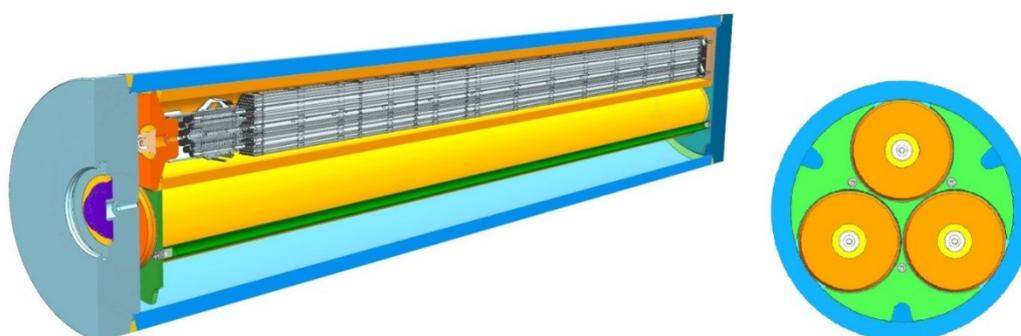
Fig. 2 ŠKODA 1000/3 WDP (Forman et al., 2021)

2 The concept behind the conducting of corrosion experiments in the Bukov URF environment

2.1 Characterisation of the selected gallery at the Bukov URF and the selection of locations for the boreholes

The geological characterisation of the ZK-3J test gallery and the surrounding area will be conducted in a specially-selected part of the Bukov URF. The resulting documentation will focus both on the structural description of the gallery, i.e., the occurrence, extent and relationship of faults and fracture systems, and a petrographic description, i.e., the representation of the various rock types and the description thereof. Based on the field data obtained, a 3D geological model of the gallery will be compiled in MOVE software, version 2020.1 (Petroleum Experts) on the basis of a photogrammetric model created in the Agisoft Metashape program (Agisoft LLC). The 3D model will include the plotting of the various documented courses of the fractures, faults and foliations and the ends thereof, as well as the range of the various rock types present. The failures identified will be extrapolated beyond the gallery so as to provide an idea of the extent of the failure of the adjacent rock mass for the purposes of the planning of the drilling phase. In cooperation with the other project suppliers, a plan of the drilling work, including the visualisation of the courses of the boreholes, will be compiled based on the 3D model.

A petrographic description of the test gallery will be compiled based on the study of samples of the various rock types present using optical microscopy methods and, if necessary, electron microscopy.

2.1.1 Location and orientation of the experimental boreholes

With regard to the unavailability of data from the floor and corners of the gallery, a narrow-profile characterisation borehole will be drilled aimed, especially, at identifying the course of rock failures at depth in the rock massif. The characterisation borehole will be located in such a way that allows for the validation of the data obtained from the walls of the test chamber (verification of the course of rock failures) see Fig. 3, the minimisation of the influence on the subsequent experimental boreholes and the determination of the appropriate drilling depth. The borehole will have a diameter of 76 mm (core drilling) so as to allow for the taking of logging measurements. The depth will be determined on the basis of the 3D structural model and the requirement to verify the various structural elements (provisionally proposed depth of approx. 12 m).
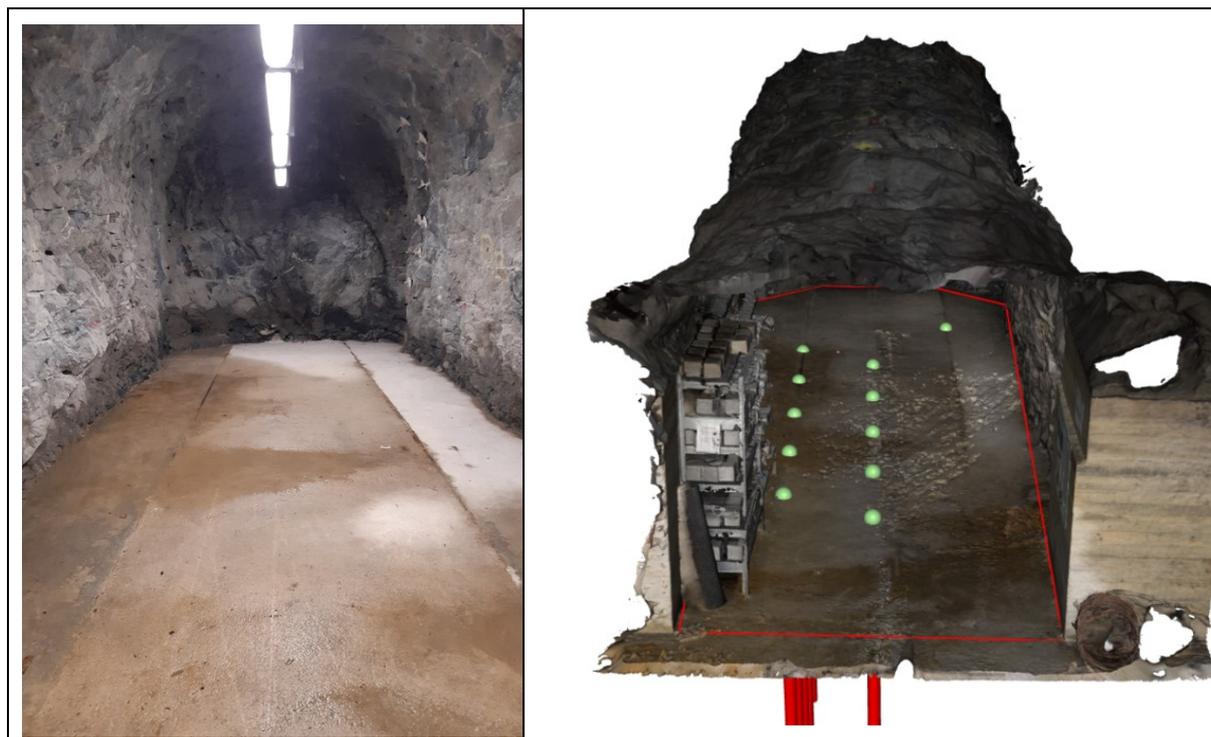
Fig. 3 a) Experimental chambre, b) 3D gravimetric model

Selected logging measurements will be taken (using e.g., a television camera, ABI, OBI, inclinometry, cavernometry) so as to determine the orientation of the faults. Following the evaluation of the ABI/OBI data, the fault orientations identified will be plotted in the 3D structural geological model and validated with data obtained from the walls of the ZK-3J chamber.

A further aim of the characterisation borehole will concern the verification of the methodology for the cleaning, pumping and minimisation of the microbial contamination of the borehole (sterilisation and instrumentation). Following the drilling of the borehole, the drill core will be studied and documented followed by the cleaning phase (sterilisation of the borehole) and the renewal of the natural groundwater (repeated pumping, groundwater renewal, stabilisation of the conditions).

Following the cleaning of the borehole and the renewal of the natural groundwater, hydrochemical profiling will be performed aimed at the monitoring of the conditions in, and the possible flow of water into (conductive structures), the borehole. Measurements will also be taken of the rate of the stabilisation of the conditions, especially the measurement of the oxygen content (O_2) and the oxidation - reduction potential (ORP). Electrochemical probes will be installed to a selected depth so as to allow for the long-term recording of the various monitored parameters.

Following the characterisation and assessment of the environmental conditions, the locations of the boreholes will be determined based on the selected criteria. In addition, the weighting of the various criteria will be evaluated with regard to the objectives of the project. It should be noted that some of the criteria and requirements are in conflict. The distance and geometry between the boreholes will be chosen so as to ensure both similar conditions in all the experimental boreholes (for comparison purposes) and that the samples are not mutually

influenced. Therefore, the introductory part of the experiment will include the creation of a basic thermal heat dissipation model and research focusing on the mutual and overall influence of the studied modules. It will then be necessary to determine the appropriate layout based on the determination of the experimental priorities according to the requirements set by the SÚRAO.

Main requirements:

- Suitability of conditions - oxygen content below 1%
- Similarity of conditions - ie as similar conditions as possible in individual wells - wells as close to each other as possible (similar hydrochemistry, microbiology, etc.)
- Non-influence: mutual non-influence of individual materials with each other - distance of boreholes from each other min. 70 cm
- Variability: possibility to decide / select suitable wells based on characterization and specific conditions. Possibility to add other wells in a similar geometry in case of well collapse or unsuitable natural conditions

The main assessed criteria:

I. Microbiology: most similar starting conditions in boreholes to compare microbial communities during the experiment. Also microbially influenced corrosion is serious issue especially at the beginning of the emplacement, when this phenomenon can cause serious local penetration into the metal surface, due to a direct electron transfer between bacteria and metal.
I. Geochemistry – hydrochemistry: most similar starting conditions in boreholes to clarify changes during the experiment
II. Geology - structural geology: minimize communication between boreholes to prevent interaction between different bentonites, maximum distance from the main faults
III. Influence of temperature: minimize effect of heating on empty boreholes to allow possible insertion of non-heated modules
IV. Technical/technological criteria: allow easy installation, sampling, manipulation, access, dismantling etc.

The outcome will comprise the determination of suitable locations, geometries and depths for the 11 planned boreholes.

2.1.2 Excavation of the experimental boreholes

Based on the characterisation of the test chamber and its surroundings, the results of measurements taken in the characterisation borehole and the evaluation of the other criteria (technological, microbiological, geology, geochemical, thermal) various options concerning suitable positions for the experimental boreholes will be identified and presented to the SÚRAO. The experiment will involve the study of 11 boreholes with diameters of between 90 and 250 mm, depending on the final design of the corrosion modules (following approval by the SÚRAO). The diameter will be chosen with regard to the safe immersion of, and the required volume of water surrounding, the modules and the requirements of the dismantling phase.

The depth of the boreholes will depend on the geological (structural) and hydrochemical conditions. Following the drilling of the 76 mm-diameter characterisation borehole, it will be

thoroughly cleaned, the natural groundwater will be renewed and the borehole will be sealed (using an obturator). Repeated hydrochemical profiling and microbiological sampling will subsequently be performed for the monitoring of the development of natural conditions in the borehole. The decision on the depth of the experimental boreholes will be based on the results of the monitoring of the hydrochemical parameters in borehole S-18 (from the ZK-3S gallery), from measurements taken in the characterisation borehole (hydrochemistry, logging, etc.) and the geological characterisation of the gallery. The conditions will then be validated with the results of the hydrochemical measurements taken in the characterisation borehole (76 mm).

The drilling work will proceed on the basis of the "Excavation of test boreholes in the ZK-3J gallery" project documentation, and will include a description of the site, the drilling process and related requirements, the drilling technology and the other technologies employed, the labelling system, the orientation of the drill cores, the obligations of the various suppliers, etc.

The drilling procedure will comprise core drilling using diamond drill bits (standard diameters) with water flushing. Prior to drilling, the instrumentation will be thoroughly cleaned and disinfected. Anaerobic groundwater from the S-1 borehole will be used for cooling and flushing purposes; the composition of S-1 groundwater reflects the composition of that in the Bukov URF and is available in sufficient quantities. The water will be pumped into a prepared tank and supplied via a flow filter to cool the drill bit. Prior to the commencement of the drilling work, the drill bits/pipes will be cleaned using pressurised water containing a sterilising agent. Emphasis will be placed on the cleanliness and accuracy of the drilling work aimed at minimising the potential contamination of the boreholes.

The drilling work will be fully supervised and a detailed drilling log will be maintained according to the SÚRAO quality standards and procedures (template). The drill cores will be placed in core boxes with maximum accuracy, formatted and documented in detail (see chapter 2.2.1).

Following the drilling process, the boreholes will be thoroughly cleaned and the borehole walls disinfected (to prevent microbiological contamination that would increase dead DNA in water), and the groundwater will be drained repeatedly from the boreholes thus the boreholes will be cleaned of disinfection. They will then be left for the stabilisation of conditions and natural filling with fresh groundwater.

2.2 Characterisation of the boreholes

2.2.1 Geological characterisation

Following the drilling of the cores, they will be documented in detail and a drilling log will be created for each core. Where possible, the cores will be scanned using a core scanner (DMT Core Scan 3). The drilling log will be created using specialised software (LogPlot, CoreScan or WellCAD). Each drilling log will contain an image of the whole of the drill core, the documented fractures (drilling-induced and natural) and faults including the fillings thereof and the representation of the various rock types, for the purposes of the description of which samples will be taken from the drill cores to allow for the more detailed determination of their characteristics using petrological sections, which will be studied via optical microscopy and supplemented by electron microscopy if necessary. The data obtained will concern principally the evaluation of the geological phenomena of the boreholes, the detailed description of the detected rock types and the characteristics of structural features identified.

Following the drilling of the characterisation borehole (76 mm), borehole logging (ABI/OBI television) will be performed aimed at determining the main rock failures (natural fractures or faults) in the borehole. The 3D geological model will be validated based on the results, and proposals for the locations and depths of the 11 experimental boreholes will be submitted.

2.2.2 Evaluation of the conditions in the boreholes

Following the drilling and cleaning of the experimental boreholes intended for experimental modules (1-10, note: borehole 11 will be used primary for water monitoring), the groundwater levels and water temperatures of, as well as the flows of water into, the boreholes will be determined. Based on hydrochemical profiling (using a multiparametric probe), the conditions and vertical composition of the groundwater (Eh, O_2, pH, conductivity) will also be determined. The hydrochemical profiling will then indicate the depths, e.g. fault zones, for the sampling of the groundwater for the hydrochemical analysis. Inflows to individual wells will be monitoring before packing and after installation of the packer system.

The characterisation phase will also include the collection of microbiological samples for the monitoring of potential contamination and the study of the development of microbiological communities. Samples will be taken under sterile conditions from selected depths using vacuum filtration (see chapter 4.10).

Following the basic sampling campaign, the boreholes will be sealed using mechanical obturators (packers) and left for the hydrochemical conditions to stabilise. The development of O_2 and Eh conditions in the boreholes will be monitored on a regular basis. Groundwater samples will be taken during the stabilisation phase for the monitoring of the basic hydrochemical parameters. Particular attention will be devoted to the testing of the influence of groundwater sampling during the defined closed intervals on changes in the hydrogeological and hydrochemical conditions and the reaction of the microbiological communities. The test results will then allow for the optimisation of the sampling plan aimed at minimising the impact of sampling on the stability conditions and the maintaining thereof.

Based on the results of the groundwater analysis and the measurement results, the conditions in the boreholes will be assessed and, if necessary, the measurement campaign will be repeated. The assessment will be performed based on the physico-chemical parameters and with regard to the development of microbial activity in the boreholes. The temporal development will be discussed on a regular basis inside expert teamso as to be able to adequately respond to the development of the situation. The aim will be to attain a steady state system. Should it become clear that significant changes are still underway within the system and that there is a risk of exceeding the set project deadlines, the subsequent development of the experiment will be resolved inside expert team.

According to contract SO2020-086, the experiment will be terminated if the oxygen concentration in the groundwater of the boreholes exceeds 1% in the steady state and fails to decrease over time.

A summary of the results will be provided in the Characterisation of a gallery at the Bukov URF report.

2.2.3 Characterisation of the groundwater prior to the installation of the experimental modules

The characterisation of the groundwater will be performed prior to the installation of the experimental modules in order to determine the electrochemical parameters of the pH, redox potential, conductivity and residual oxygen, the value of which must be below 1% prior to installation. In addition, groundwater samples will be taken for the hydrochemical and microbiological analyses for determining the initial state of the system. Groundwater for subsequent analysis purposes will be extracted from the depths of the expected locations of the corrosion modules so as to minimise the impact on the monitored system, i.e. the amount of water extracted will be restricted to the amounts required for analysis purposes. The preparation of the groundwater extraction equipment will follow the set microbiological requirements, see below. The microbiological analysis will include microbiological depth profiling in the characterisation borehole and additional sampling in connection with the microbiological characterisation of the gallery environment. A total of 10 samples will be assessed, of which 7 samples from the characterisation borehole and 3 samples from the surrounding groundwater sources. The first sample will be taken from the characterisation borehole immediately following cleaning and the draining and refilling of the groundwater in the borehole. In order to minimise undesirable microbial contamination, water from S1 borehole will be used during the drilling and cleaning of the characterisation borehole; moreover, all the samples will be taken using thoroughly sterilised equipment (chemically - FreeBact, Xinix or autoclaving), which is essential for the subsequent microbiological analysis. The conditions in the borehole will then be determined. Once the conditions have stabilised, three more samples will be taken from depths of 2, 4 and 6 metres. The borehole will then be sealed with a packer. The final sampling campaign (from the same depths) will be conducted once the oxygen level in the borehole decreases below the set level. Further details of the sampling procedure are provided in chapter 4.10. The sample processing procedure is described in chapter 4.15.3.

2.3 General description of the corrosion test

The corrosion test at the Bukov URF has been designed with the aim to verify the properties of the materials that are being considered in the Czech waste disposal package (WDP) concept, which is based on an outer carbon steel casing and inner casings of stainless steel for individual fuel assemblies. The WDP will contain three stainless steel inner casings for VVER1000 reactor fuel and seven for VVER440 reactor fuel. Thus, the concept envisages a carbon steel WDP outer casing which will provide a protective barrier during the period in which the elevated surface temperature (in connection with natural chloride content in bentonite or thiosulphates provided by synergistic effect of sulfate-reducing bacteria and radiolysis) of the WDP will increase the risk of the localised corrosion of the stainless steel inner casing (Forman et al., 2021). The conditions under which the corrosion test will be performed have been chosen so that they are on the side of caution (i.e. the conservative approach will be applied): the corrosion test will take place at an elevated temperature under anaerobic conditions. The experimental environment was chosen so as to simulate the expected conditions of the future Czech deep geological repository (DGR) around 100 years following the disposal of the WDP. The corrosion samples will be placed in compacted bentonite of Czech origin and in Wyoming

type bentonite so as to provide for a comparison of the results from the two types of materials. The Czech candidate bentonite is extracted from the Černý vrch deposit and is known as BCV 2017, abbreviated to BCV (the materials sheet is provided in Appendix 1). BARA KADE® 200 mesh (the materials sheet is provided in Appendix 2), hereinafter referred to as BARA KADE, was chosen as the reference bentonite material. In addition to the steel materials intended for use in the Czech WDP concept, Cu-OF copper, which is being considered in other WDP concepts internationally, will also be tested in the experiment for comparison purposes under the same conditions as the carbon steel.

The preparation of the experiment will take place under laboratory conditions with the final phase being conducted in glove boxes where, under anaerobic conditions, the samples will be pre saturated in order to minimise the intrusion of oxygen into the modules with the samples during their emplacement in the boreholes. The transport of the sample modules to the Bukov URF will take place under argon atmosphere conditions so as to prevent the access of oxygen to the modules.

The boreholes in which the sample modules will be installed will be sealed with mechanical obturators following drilling and the groundwater in the boreholes will be monitored. The modules will be installed once the chemical and microbiological conditions have stabilised. When installing the modules, the time during which the modules are exposed to atmospheric oxygen will be minimised. The modules will be emplaced beneath the groundwater sampling devices and the boreholes will be re-sealed with the mechanical obturators. The cables and capillaries of the groundwater sampling devices will be routed through the obturators. Once the modules have been installed in the boreholes, temperature sensors and heating cartridge will be connected to a heater control panel. The temperature will be maintained at 70°C in the samples. This temperature was chosen with regard to the temperature drop over time following the period in which the dose rates on the surface of the WDP have not yet attained negligible levels. Moreover, the corrosion test is intended to simulate the anaerobic phase of the development of the disposal system with the associated elevated temperature, i.e. the aim is not to consider the temperatures of up to 95°C at the WDP/bentonite interface that are anticipated immediately following the disposal of the WDP, at which time we assume aerobic conditions will apply with the influence of ionising radiation on the disposal system.

The regulation of the temperature will be monitored remotely and will be protected against overheating via a system of safety elements. In addition, the occurrence of power outages will be closely monitored so as to allow for a timely response to such events.

The groundwater will be monitored throughout the course of the experiment at intervals that depend on the development of the conditions in the boreholes. We assume that following the launch of the experiment, the time interval will be 3 times per year, following which it will be reduced in order to minimise the impact of intervention into the system (assuming stable conditions).

The corrosion tests will last for 1, 3, 5, 7 and 10 years following the launch of the experiment, following which the respective modules will be transported under argon atmosphere conditions to glove boxes in which they will be dismantled. The metal and bentonite samples will be prepared for subsequent analysis also in the glove boxes.

The evaluation and processing of the samples will be summarised in the form of technical reports following their detailed analysis.

3 Detailed project documentation

3.1 Design of the modules

A scheme of the experimental module is shown in Fig. 4.
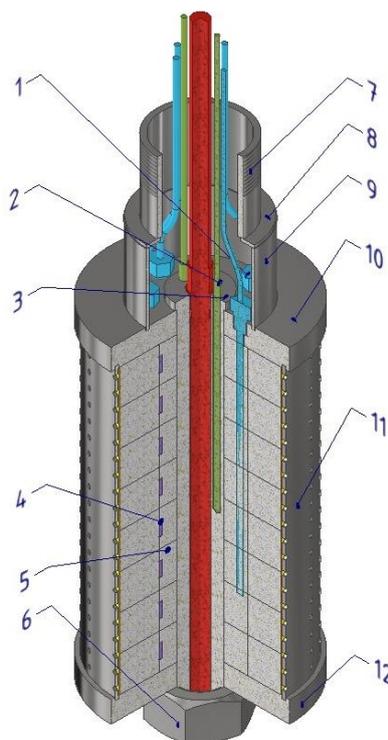
Fig. 4 Design of the module for the corrosion tests at the Bukov URF. 1 – monitoring temperature sensors, 2 – managing temperature sensors, 3 – heating cartridge tube, 4 – corrosion samples, 5 – bentonite segments, 6 – M33 nut, 7 – G2" welding nipple, 8 – connecting plate, 9 – connecting tube, 10 – cover, 11 – perforated metal sheet with coarse and fine stainless-steel fabric, 12 – bottom

All the module components will be made of stainless steel. The base element of the module will comprise a tube that will house the cartridge heater with an outer diameter of 33 mm to which a lid with a thickness of 15 mm and an outer diameter of 125 mm will be fixed. Hexagonal bentonite segments with heights of 25 mm (which will be compressed using a compacting mould) will be fitted around the central tube. The construction process will commence with the fitting of inner bentonite hexagons (width of 58 mm) into which holes will be drilled to house the corrosion samples (diameter of 10 mm and thickness of approx. 2.5 mm). This will allow for the emplacement of up to 6 samples in each level of the module. The depths of the holes will be adjusted according to the actual dimensions of the corrosion samples. Outer bentonite blocks (outer diameter of 113 mm) will then be fitted around the inner hexagons. The dimensions of the bentonite segments were chosen so that the inner faces of the corrosion samples are placed at a distance of 10 mm from the surface of the tube that houses the cartridge heater. The bentonite segments will then be covered with a fine stainless steel filter fabric (322 × 1900 mesh) that will serve to fix the bentonite in position inside the module, and then with a coarse fabric (24 × 110 mesh) that will help to evenly distribute the groundwater around the whole of the module. The outer surface of the module will then be enclosed with a sheath made of sheet metal (outer diameter of 119 mm). The bottom cover of the module (with

a thickness of 15 mm and an outer diameter of 125 mm) will then be attached to the bottom of the central tube, which will be held tightly in place using an M33 nut. The flange of the pipe housing the cartridge heater will be TIG welded or silver soldered to the lid of the module. The perimeter of the module will then be reinforced using W4 stainless steel clips and strips with a width of 12 mm to ensure that the outer casing is not deformed due to the swelling of the bentonite.

Following the assembly of the modules, holes will be drilled through openings in the lid, into which Pt100/Pt1000 temperature monitoring sensors will be inserted. Two sensor lengths (lengths of the measuring parts of 60 mm and 160 mm) will be inserted so that one of the sensors monitors the upper third of the module and the other the lower third of the module. Backup sensors (to be used in the event of the failure of the main sensors) will be emplaced axially symmetrically. The radial location of the sensors was chosen according to a pitch circle identical to that of the corrosion samples. The sensors will be sealed using silver solder.

Pt100/Pt1000 temperature control sensors with lengths of 140 mm will then be inserted into the tube (as with the main and backup monitoring sensors) along with the cartridge heater (diameter of 14.5 mm, length of 247 mm, power of approx. 300 W). A thermally-conductive paste will be applied to enhance the transfer of heat.

Following the installation of the sensors, a weldment of the nipple, the connecting plate and the connecting tube will be inserted into a specially-prepared groove in the lid and TIG welded in position.

3.2 Design of the groundwater sampling device

A scheme of the groundwater sampling device is shown in Fig. 5.
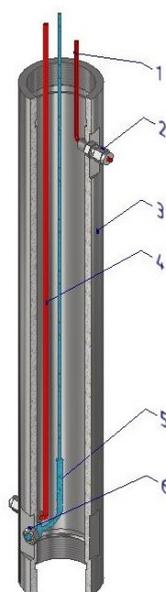
Fig. 5 Design of the groundwater sampling device. 1 – upper capillary, 2 – G1/8" bushings, 3 – sampling device tube, 4 – lower capillary, 5 – temperature sensor, 6 – M8 nut

The core of the sampling device consists of a stainless steel tube with an inner diameter of 56 mm, an outer diameter of 70 mm and a length of 400 - 500 mm. The length was chosen so as

to attain the optimal ratio of the sample taken to the volume of water in the packed interval. The tube will be equipped with G 2" internal threads at both ends so as to allow for its connection to the packer and to the corrosion module. Both threads will be sealed with lead foil due to dismantling considerations and the elimination of the use of plastics. G 1/8" threaded bushings will be installed in the wall of the tube for the connection of the capillaries, which will be sealed with silver solder following connection to the sampling device. The capillaries will then be fitted and sealed using washers. The upper capillary will be used to supply a pressurized gas and the lower capillary to drain the sampled water. Both capillaries will be equipped with valves to ensure they do not leak between the sampling campaigns. In addition, an opening will be prepared in the wall of the device for the insertion of a right-angle Pt100/Pt1000 temperature sensor and an M8 connection thread, which will be fastened to the outside of the device using a nut. The nut will then be secured in place using silver solder.

Illustrative picture of module and sampling device connection with packer is shown at Fig. 6.
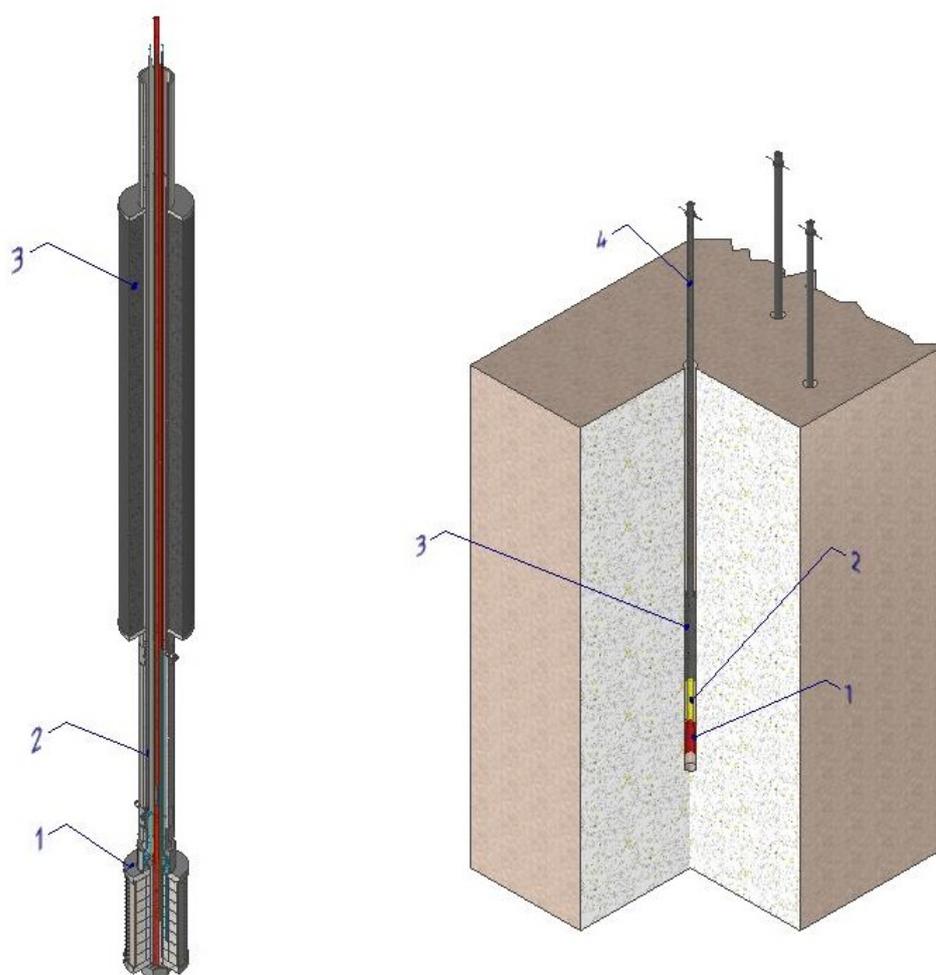
Fig. 6 Schematic picture of experimental system in host rock: 1 – module, 2 – sampling device, 3 – EPDM rubber   4 – tubes of packer system

3.3 Other components of the system

A specially-produced heater control panel which complies with Czech Mining Authority (CBU) Decree No. 22/1989, CBU Decree No. 75/2002 Coll., currently valid ČSN standards, Act No. 22/1997 and Government Regulation No. 17/2003 will serve for the regulation of the heating of the module system. The project documentation will be prepared by an electrical designer approved by the CBU. The degree of protection of the heater control panel will be at least IP 44.

The heater control panel will be equipped with ten temperature regulators with solid state relays, which will ensure the maintaining of a stable temperature in the tubes housing the cartridge heaters, i.e. a value that corresponds to a sample temperature of 70°C. The temperature control of the samples will be provided by the afore-mentioned independent temperature sensors. The switching on/off of the cartridge heaters will be regulated by semiconductor relays activated by the temperature regulators. The heating system will be protected against overheating and, in the event of a fault, the heating will be switched off automatically and will have to be restarted manually.

Furthermore, the heater control panel will feature a built-in power outage regulation system that ensures that such outages are detected in time and that the time over which the system is unheated is minimised. The control panel will also include a Comet temperature data logger, which will allow for the remote monitoring of the development of temperature in the experimental system.

4 Technical design of the experiment

4.1 Preparation of the metal samples

The metal samples will be made of the S355J2H + N steel material and will be supplied by Škoda JS in the form of plates with a weld (the weld was researched in detail by Škoda JS as part of the *Research and development of a disposal package for the deep disposal of spent nuclear fuel to the sample implementation stage* project). The samples will be prepared in the shape of discs with diameters of 1 cm and thicknesses of 2.5 mm. The samples will be ground using P80 sandpaper, cleaned in water using a brush, rinsed with alcohol and dried immediately. They will then be weighed on an analytical weighing scales with an accuracy of 1 µg prior to exposure and labelled via etching. Prior to weighing and exposure, the samples will be stored in a desiccator at a relative humidity of less than 25%. Twelve base material samples will be prepared for each combination of bentonite type and exposure time; 12 weld samples will also be prepared for the Czech bentonite, i.e. a total of 120 base material samples and 60 weld samples.

In addition to the S355J2H + N steel material samples, copper samples will also be placed in the BARA KADE bentonite for comparison purposes, specifically Cu-OF copper (EN1652/1997), which was tested in the afore-mentioned Škoda JS project. The samples will be cut from sheet metal supplied by Škoda JS and will also comprise discs with diameters of 1 cm but thicknesses of just 1 mm (the thickness of the copper sheet). The preparation of the samples prior to experimentation will be identical to that of the steel samples. Twelve samples will be prepared for exposure in the BARA KADE bentonite for each of the exposure times, i.e. a total of 60 samples for the five modules.

4.2 Preparation of the bentonite

Both the BCV and BARA KADE bentonites will be homogenised; this will be followed by the determination of their natural moisture content, based on which corrections will be made to the weights for the compacting of the segments. The required dry density of the segments is 1600 kg m^{-3} following swelling to the dimensions of the modules. This value was chosen according to experience obtained from previous experimental research. Compacting will be performed uniaxially in a hydraulic press without the use of lubricants.

Since the compaction of the bentonite will be required to ensure sufficient clearance for the assembly of the modules, the bentonite will be compacted into smaller volumes than the actual dimensions of the modules, so that the real dry density value of the compacted materials will be greater than the required 1600 kg m^{-3}. The required value will be reached only after the bentonite swells to fit the dimensions of the modules.

The bentonite materials will be compacted in the form of outer and inner hexagonal segments, see Fig. 7. The cavities for the samples will then be milled into the sides of the inner hexagons. Thus, each of the inner segments will contain 6 samples per 2.5 cm of the height of the bentonite, resulting in the stacking of 12 samples of the same type on top of each other on two levels. Two segments of the respective bentonites without samples will be positioned between the samples, i.e. the total distance between the samples will be 6.5 cm.
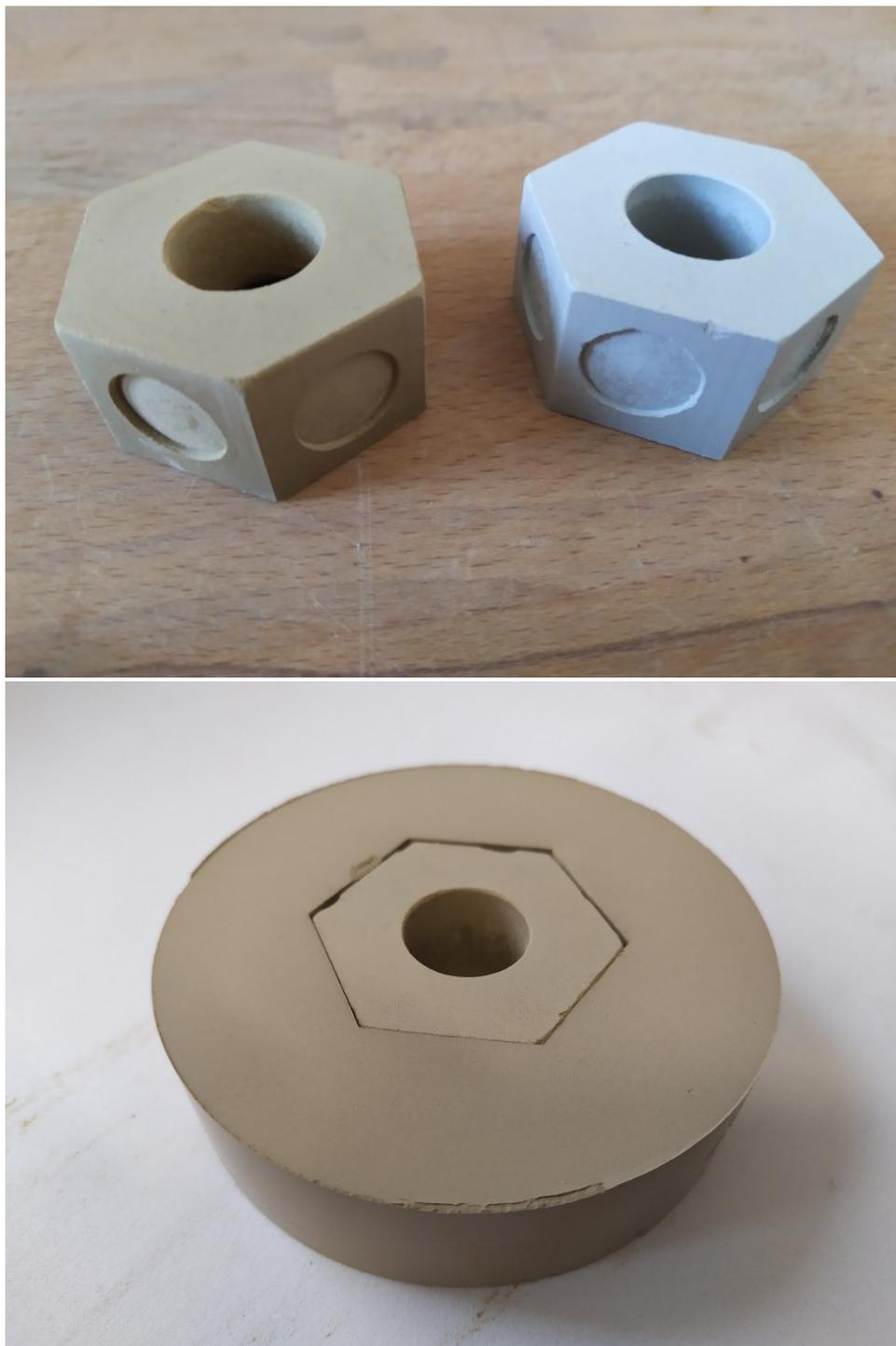
Fig. 7 Illustration of the compacted bentonite segments. Above: inner hexagons (left BCV, right BARA KADE) and below: an outer bentonite block with an inner hexagon

4.3 Plan for the input characterisation of the tested materials

4.3.1 Characterisation of the metal samples

The characterisation of the two metal materials will involve the preparation of a number of samples for the determination of the microstructural condition of the S355J2H + N carbon steel

and the copper. Samples will be taken in all 3 perpendicular planes so as to determine the isotropy of the microstructure. The samples will be encapsulated in acrylic resin, ground up to the level of P1200 grit sandpaper and then polished with a 3 µm diamond paste. Following washing, the steel samples will be etched with 2% Nital (nitric acid in ethanol) and the copper samples with a ferric chloride solution (5 g of ferric chloride, 50 ml of hydrochloric acid and 100 ml of water) and the microstructures observed using a metallographic monocular microscope.

Original roughness of the samples surface will be specified by means of mechanical profilometry. Three representative samples of each material will be scanned.

The elemental composition of the materials will be verified by means of X-ray fluorescence spectroscopy (XRF) and optical emission spectrometry (OES). The samples will also be analysed via X-ray diffraction (XRD) and scanning electron microscopy with an energy dispersive elemental analysis (SEM/EDS). Small carbide particles will be identified via phase diffraction analysis employing transmission electron microscopy (SAED HR-TEM).

Surface analysis before exposure will be employed. X-ray photoelectron spectroscopy (XPS) will be used for identification of surface composition of the specimens before and after presaturation of the bentonite segments. XRD and Raman spectroscopy will be employed as well for the after-presaturation specimens.

4.3.2 Characterisation of the bentonite

It was considered advantageous to characterise the selected bentonite materials prior to their installation in the in-situ experiment. By comparing the results with the analysis that will be performed of the bentonite materials after the various planned exposure times of the in-situ experiment, it will be possible to determine the changes that occurred in the bentonite. This section provides a summary of the methodologies that will be applied in the initial characterisation of the two types of bentonite; chapter 4.15.2 subsequently outlines the plan for the analysis of the bentonite samples taken from the in-situ experiment. The expected results on the behaviour of the bentonites under in-situ conditions are outlined in chapter 5.2.

The following methodologies were selected for the initial characterisation of the bentonites, which are described in more detail for reasons such as different approach in the procedure, the existence of alternative methods that would provide the same results, etc.:

- X-ray fluorescence spectroscopy (XRF)

Powder X-ray fluorescence spectroscopy is a simple and rapid method for the determination of the elemental composition of materials. The chemical composition can be expressed as the mass content of individual elements or in the form of ideal oxides.

- Determination of the content of carbon and sulphur species (TOC, TIC and TS)

From the point of view of corrosion, organic carbon is a species of interest because it has the potential to serve as a nutrient for microbially-induced corrosion, as does sulphide or sulphide that has been metabolically processed via microbial activity from sulphate that causes so-called sulphidic corrosion. The carbon species content will be determined by combusting a sample at high temperatures while monitoring gaseous products using the appropriate analyser.

- Thermal analysis with the analysis of evolved gases (TA-EGA)

Thermal analysis with the analysis of evolved gases is complementary method to the determination of the content of carbon and sulphur species. It provides comprehensive information but without the quantification of the various components. The heating of the powdered material in an Al_2O_3 crucible up to 1000 °C in a normal or argon atmosphere provides information on weight changes (the outputs are TG or dTG curves), temperature changes between the sample and the standard during heating (the output is the DTA curve) and the evolution of gases (e.g. H_2O, CO_2, SO_3, etc.) using the coupled mass spectrometry.

- Powder X-ray diffraction analysis (XRD)

At this stage, powder X-ray diffraction analysis will be performed in order to determine the semi-quantitative compositions of the materials only. More detailed analysis aimed at the detailed determination of the expandable structures will be performed during the analysis of samples extracted from the in-situ experiment.

- Analysis of leachable ions

The initial characterisation phase will include the analysis of leachable ions under aerobic conditions. Naturally moist (initial water content) bentonite powder of a known moisture content will be mixed in various phase ratios with deionised water (from a min. of 20 to a max. of 400 g/L). The suspensions will be shaken for 28 days. Following the centrifugation of the suspensions, the supernatants will be collected, filtered through a 0.22 μm membrane filter and subsequently subjected to the determination of the major ion concentrations and the electrochemical parameters (pH, electrical conductivity).

- Cation exchange capacity (CEC)

The cation exchange capacity (CEC) is a good indicator of the montmorillonite content (unless another cation exchanger is present in the material). The CEC will be determined using certified methodology (Červinka 2014) using Cu(II)-triethylenetetramine. The outputs will comprise both the CEC values derived from the decrease of the amount of copper (with the index of the determination method, CEC_VIS using spectrophotometry and CEC_AAS using atomic absorption spectrometry) and the sum of the displaced cations (the so-called CEC_sum), as well as the representation of individual cations in the montmorillonite exchange positions. The monitoring of the difference between the various CEC values is also important to the evaluation. Higher CEC_sum values than the CEC_VIS or CEC_AAS may indicate the presence of soluble mineral phases. The amount and composition of such phases are determined via the analysis of the leachable ions (see above).

As mentioned by Svensson et al. (2019), the repeatability of the CEC method using Cu-triene is adequate but with less good reproducibility of results (measured on different occasions). It is recommended to analyze all the samples to be compared in one set. Since the CEC determination is not demanding, this approach (Svensson et al. 2019) will be chosen in this project as well. The input material will, in all cases, be measured when measuring the samples from the in-situ experiment, also in the case of the determination of:

- Specific surface area (SSA)
- Swell index (SI)

These determinations, as the CEC determinations, will be performed in a set with real samples taken from the in-situ experiments. More detailed descriptions of the two determinations are provided in chapter 4.15.2.

- Microbiological characterisation

The bentonites used in the experiment will be microbiologically characterised in advance via the molecular-genetic analysis of the DNA, in the same way as described in chapter 4.15.2., according to the study of Engle et al. (2019). All bentonites (powder and compacted) will be processed in the same way, but while with the compacted we do not expect insufficient recovery and DNA yields during isolation, this can happen here. Should the direct isolation of the DNA from the supplied bentonite powder prove to be insufficient (too low a DNA yield), the screening of the microbial activity in natural bentonite will be performed using a short-term culture in the suspended state so as to detect the degree of microbial activity. It is important to note that we are concerned with answering a different question for powder and compacted bentonites - total potential for microbiome revival (powder bentonite) and current microbiome revival at the end of the experiment (compacted bentonite).

4.4 Preparation of the modules

The assembly and completion of the modules will take place under aerobic and laboratory conditions. The first phase will involve the welding/soldering of the metal joints between the modules and the sampling devices so as to ensure their tightness. This will be followed by the placing of the compacted bentonite blocks with the samples in the middle parts of the modules; once installed, they will be covered with filter cloths (fine and coarse). The outer casings will then be fitted around the modules and the system will be fixed with a lid and a nut. Holes will subsequently be drilled into the closed bentonite modules for the emplacement of the temperature sensors. The connections between the temperature sensors and the module covers will then be soldered so as to ensure the tightest fit possible.

The assembled modules will then be transferred to glove boxes, where they will be left for at least 7 days to allow for the removal of residual oxygen, following which they will be immersed in deoxygenated demineralised water for at least 14 days (Dobrev et al 2011). The use of demineralised water for saturation purposes was considered ideal in view of the low salinity of the future host groundwater at the Bukov URF. Indeed, the results of extensive research on the transport of species in saturated compacted bentonite indicate that the composition of the bentonite pore water is not significantly affected by the solution used to saturate the bentonite (Hofmanová et al. 2019). Following the saturation period, the modules will be packed in sealed bags and transferred from the glove boxes to argon-filled transport containers.

Thus, 5 BCV bentonite modules with carbon steel and weld samples and 5 modules with BARA KADE bentonite with carbon steel and copper samples will be constructed. Each module will contain 12 samples of each type of metal materials, i.e. a total of 24 samples per module.

4.5 Transport of the modules to the Bukov URF

The modules will be transported in sealed transport containers filled with argon with slightly elevated pressure. The transport of the containers will be planned in accordance with the module installation plan, which will depend mainly on the timing of the saturation of the modules.

4.6 Plan for the emplacement of the modules in the boreholes

The timing of the emplacement of the modules in the boreholes depends on the fulfilment of the set condition of a maximum oxygen content in the boreholes of 1% (according to contract SO2020-086). Furthermore, the stabilisation of the groundwater in the boreholes will be monitored in terms of its chemical composition, electrochemical parameters and the microbial communities present. The emplacement process is required to take place under relatively stable system conditions. In the event that any significant changes occur in the system in the period up to the agreed deadline for the fulfilment of this part of the project schedule, further progress will be resolved with SÚRAO bearing in mind the status of the system, i.e. the decision will be made jointly whether to postpone the emplacement of the modules or to install them within the original time schedule.

Prior to the emplacement of the modules in the boreholes, the heater control panel will be installed and put into operation. The modules with the samples will then be emplaced in the boreholes. The first phase will include the sampling of the groundwater, which will be followed by the removal of the mechanical obturators with the sampling devices, which will subsequently be fixed to the sample modules as quickly as possible so as to minimise the time during which the modules are exposed to atmospheric oxygen.

The various cables will be routed via the mechanical obturator in protective tubes so as to prevent damage to the cables during the emplacement of the modules in the boreholes. Only one borehole will be opened at any given time, following the closure of which the next borehole will be opened. The modules will be protected from dropping accidentally into the boreholes via the use of self-locking pliers with a chain at all times.

Following emplacement in the boreholes, each of the systems will be filled with argon in order to verify the effectiveness of the sealing of the mechanical obturators. If leaks are detected, the mechanical obturator will be tightened. Finally, the system will be flushed with argon through the sampler so as to create a slight argon overpressure of up to 0.5 bar. The opening of the packer system will be closed using a bushing that allows for the filling of the inner part of the packer with argon so as to prevent the penetration of O_2.

Once the temperature sensors and the cartridge heaters have been connected to the heater control panel, the functionality of the whole of the system will be verified.

4.7 Launch of the experiment

The conducting of the long-term corrosion experiments is planned for 1, 3, 5, 7 and 10 years. The experiments will commence following the emplacement of all the modules in the respective boreholes. Initially, the modules will be heated to around 40°C in order to verify the integrity of the experimental systems. It is envisaged that this temperature will be sustained at the position of emplacement metal samples in bentonite so as to allow basic thermal stabilisation of the system for around 24 hours. The temperature will subsequently be gradually increased up to the target of 70°C at the position of the samples (the temperature in the middle of the heater will be increased further to ensure that the target temperature is maintained at the position of the samples depending on the behaviour of the system). The experiment proper will commence once the systems have stabilised at 70°C. It can be assumed that within a few days of the start of the experiment a slight shift will occur in the temperature in the samples due to

ongoing system stabilisation (a reduction in the degree of bentonite saturation near the heater, an increase in the temperature around the modules, changes in the temperature of the rock in the vicinity of the modules) that will require the adjustment of the heater temperatures. The overall stabilisation of the systems is expected to occur several months following the launch of the experiment.

The monitoring of the temperature for the remote control of the experiment will commence following the emplacement of the modules.

4.8 Plan for the operation and maintenance of the experiment

The experiment will be conducted in the humid environment of the Bukov URF and will be visually inspected on a regular basis during the extraction of groundwater. The following issues will be subjected to regular verification: the condition of the equipment, including the heater control panel, with regard to potential corrosion and the pressure in the system via the verification of the tightness of the mechanical obturators. The condition of the various electrical components of the equipment will also be checked and, if necessary, the affected components will be replaced.

Only semiconductor relays will be used in the experiment due to their better service life than that of mechanical relays. Based on experience gained from the MaCoTe project, where problems with the mechanical relays were detected after around three years of the running of the experiment, the replacement of the semiconductor components in the Bukov corrosion experiment will be considered after five years of experimentation.

The system will provide for the monitoring of power outages, which will enable the control of such events from outside the Bukov URF complex (i.e. remotely). In the event of a power failure, the heating system will be set so that it does not start heating once the power supply is restored, i.e. the heating of the modules will have to be restarted manually. This method was chosen due to previous experience with temperature control failures following power outages which led to the unregulated heating of the system. Since the occurrence of such failures is usually unpredictable, in terms of the effect on the experimental system, it is better not to heat the system than to risk uncontrolled heating, which would lead to the total loss of the experiment. In addition, the system will be equipped with a fuse to prevent uncontrolled heating, which will automatically switch off the heating system if the set limit temperatures are exceeded.

4.9 Monitoring of the experiment

The experiment will be monitored via the remote checking of the temperatures at various positions in the samples, and will continue until the end of the experimental period. In addition, the extraction of groundwater samples will continue throughout the experiment and monitoring of pressure under packer systems will be performed. Any problems that occur will be solved as a matter of priority.

4.10   Monitoring of the groundwater

The groundwater will be monitored in all 11 boreholes for a period of 12 years following the drilling work. According to contract SO2020-086, the number of sampling campaigns is set at three per year with the potential for reducing them to two or one per year should similar results be obtained three or five times in a row, respectively. In addition to the conditions for reducing the number of sampling campaigns defined in the contract, it will also be necessary to take into account the influence of the extraction of groundwater on the system. Should an increase in the oxygen content be identified following the extraction of groundwater due to the transport of oxygenated water through the fracture system, it will be necessary to consider a reduction in the number of sampling campaigns for the respective borehole.

Sampling will be performed in the first phase following the drilling of the boreholes using standard sampling equipment designed for use in underground environments. This will be followed by the installation of sampling devices in the boreholes produced in accordance with the technical documentation. These sampling devices will be used for the monitoring of the groundwater up to the end of the project.

The sampling devices work on the principle of the expulsion of water from the borehole using argon and a dual-capillary system (one capillary for the supply of argon and the other for the extraction of the groundwater). The measurement of the electrochemical parameters during the extraction of the groundwater will involve the use of a special modular cell, which allows for flushing with argon and its subsequent filling with groundwater. The cell can be modified according to the number of electrodes required and was subjected to detailed testing as part of the MaCoTe project (Dobrev et al. 2021). Groundwater samples for the chemical analysis will be taken in special plastic sample boxes supplied by a certified laboratory. Groundwater samples will also be taken for the determination of the presence of colloidal particles.

Slightly pressurised argon will be left in the boreholes following sampling. The groundwater monitoring results will be summarised in the form of a technical report in line with the requirements of the project schedule; each subsequent technical report will comprise the revision of the previous report.

The hydrochemical parameters of the characterisation borehole (76 mm) will also be monitored regularly using am MS5 hydrochemical profiling multiparametric probe.

In order to be able to obtain microbiologically relevant data, it is absolutely essential that the groundwater samples be taken from packed anaerobic flooded boreholes. Due to the serious risk of microbial contamination, it is necessary to minimise the associated risks by following a number of basic recommendations – the use of water from surrounding boreholes during the drilling process, the thorough cleaning of the drilled borehole before installing the packers and, finally, the use of only sterilised instrumentation for monitoring and sampling purposes.

It is assumed that approximately 0.3 -1.5 l of water will be taken from each borehole in sterile glass sampling bottles (1 l). The groundwater samples obtained will be immediately filtered on site using a sterile disposable Omnifix 50 ml syringe (BBraun, Switzerland) fitted with a sterile Sterivex filter (0.22 µm, Millipore, Germany). This methodology has already been tested and is routinely employed in the MaCoTe project (Dobrev et al. 2021). Since the use of Sterivex filters is optimal for prescribed volumes of water (Brazelton et al. 2012; Casar et al. 2021; Miettinen et al. 2015; Nyyssönen et al. 2014; Robador et al. 2016), the exact filtered volume

must be recorded for each sample; this information is important in terms of subsequent data processing (qPCR).

Filtration using the Sterivex filter allows for the capture of the spectrum of native microorganism species present in groundwater samples. Moreover, the use of the filter will subsequently allow for a significant reduction in the volume of material considered in the microbiological analysis, and will generally simplify its handling during transport and the subsequent isolation of DNA. However, the minimisation of potential contamination with other microorganisms comprises one of the most important factors when extracting water with low concentrations of microorganisms (which is typical of groundwater). The Sterivex filters with the captured microorganisms will be placed in resealable sterile PE bags, labelled and stored in a portable cooling box between cooling blocks at a temperature of up to 10°C immediately following the filtering of the samples. The filters will be stored and transported on the same day as the collection of the samples and transferred to a freezer with a set temperature of -80°C. They will remain under such conditions until the DNA isolation phase.

4.11 Plan for the removal of the corrosion modules

The modules will be removed 1, 3, 5, 7 and 10 years following the launch of the experiment. One BCV and one BARA KADE bentonite module will be removed after the given time intervals. The selection of the location of the sampling of the modules will depend on the conditions in the boreholes following stabilisation and taking into account efforts to minimise the influence of the vacated boreholes by the other boreholes in which the experiment continues.

At present, it is assumed that the modules with the longest sampling times will be emplaced in those boreholes that are least likely to be affected by the extraction of groundwater, i.e. those with the most stable conditions. Conversely, the one-year experimental modules will be emplaced in those boreholes that are most prone to changes in their conditions due to groundwater extraction. However, this aspect can only be considered following the completion of the first phase of the groundwater monitoring prior to the emplacement of the modules, as well as with regard to the possible expansion of the project, which SÚRAO can request according to the terms of the contract. If the project were to be expanded, spare boreholes, which have been rated of the "worst" quality, would be available for the conducting of the longest-term corrosion tests.

Assuming the expected development of conditions in the boreholes and the launching of the experiment in 2022, the modules will be removed from the boreholes in 2023, 2025, 2027, 2029 and 2032.

Groundwater will be extracted for the determination of the electrochemical parameters prior to the removal of the modules. When extracting the assemblies, they will be protected at all times from accidentally falling back into the boreholes. Following the removal process, the module with the corrosion samples will be separated from the experimental assembly and the sampling device will be left in the borehole for the continued monitoring of the groundwater; the end of the device will be closed to prevent the penetration of oxygen into the system. The modules will be placed in argon-filled transport cases immediately following their removal. The rest of the assembly will then be re-installed in the boreholes. Finally, the sampling device will be flushed with argon to a slightly elevated pressure according to the local conditions.

4.12   Transport of the modules to the laboratory

The modules will be transported to the laboratory in hermetically-sealed containers filled with argon under elevated pressure. The containers will be flushed with argon before the modules are placed within them. They will then be closed and flushed again with argon via a valve. Finally, under an elevated argon pressure, the containers with the modules will be transported to the laboratory for dismantling in glove boxes.

4.13   Dismantling of the modules in the glove box

The first step before the dismantling of the modules in the glove box will comprise preparations for the microbial sampling, where, again, it will be essential to minimise the risks of contamination. The preparation process will consist of sterilising both the tools required and the glove box using a germicidal lamp with the thorough wiping of all the surfaces (detergent and ethanol). The next step will be to place the modules in the glove box via a transition chamber. This will inevitably involve the exposure of the modules to atmospheric oxygen for a short period of time. There are two ways available for removing oxygen in the transition chamber; the first method involves the evacuation of the transition chamber and its refilling with argon - this cycle is repeated several times at a vacuum pressure of 0.6 (a higher vacuum pressure level would significantly increase the degree of water evaporation). The second option is to flush the transition chamber with argon for a certain period of time; while this approach would ensure less water evaporation from the modules, it would mean the less efficient removal of the residual oxygen. From a general point of view, we assume the partial evaporation of water from the filter fabrics surrounding the modules in the transition chamber; however, a decrease in the moisture content of the bentonite surface layer cannot be ruled out. Due to the minimal humidity levels in the glove boxes, however, the modules will be dried immediately following their placement therein; thus, the influence of the humidity of the bentonite due to exposure in the transition chamber will be negligible compared to the changes in the glove box. Nevertheless, the preferred option includes the availability of an evacuation route from the transition chamber aimed at minimising the amount of oxygen in the system.

The placing of the modules in the glove box (only one module will be present in the glove box at any one time) will be followed as quickly as possible by the start of the dismantling process. Provided no technical or other unexpected complications arise, the outer cover and the filter cloths will be removed from the module in a relatively short time, thus allowing for the transfer of the bentonite to sealable sample boxes for the determination of the moisture content. However, it is important to note that no matter how quickly the outer cover and the filter fabrics are removed, the bentonite samples will still be partly dried in the glove box, which will inevitably influence the determination of the moisture content.

The dismantling of the modules will continue with the removal and identification of the metal samples and the taking of bentonite samples for chemical and microbial analysis purposes from the outer parts of the bentonite, the areas surrounding the metal samples and the vicinity of the heater. The samples will be carefully labelled and photographed.

4.14   Preparation of the samples for analysis

The metal samples will be rinsed with alcohol and left in the glove box until the residual moisture has been removed from the surfaces (the drying time will be at least 1 week). This method was chosen so as to minimise the oxidation of the corrosion products following contact with the air atmosphere outside the glove box. The samples will then be transferred in sealed sample boxes for the subsequent analysis phase. No further modification of the samples will be necessary for the chemical analysis. The size of the samples will be adapted for further analysis purposes.

The samples of bentonite intended for the chemical analysis will also be left in the glove box to dry; the drying time will be selected on an ad-hoc basis according to the size of the sample and expert estimates of the required drying time depending on the specific conditions of the samples. Alternatively, the samples may be dried in an integrated glove box dryer. Although the samples will then be placed in sealed sample boxes, they will still absorb a certain amount of air humidity due to contact with the atmosphere, which may lead to partial oxidation-initiated changes. Therefore, the exposure time of the samples to the atmosphere prior to the analysis phase will be minimised.

The bentonite samples intended for the microbial analysis will be frozen in sterile bags immediately following their collection and transferred for analysis. In view of the thicknesses of the bentonite rings in the experiment, bentonite samples (of an optimum weight of 5 g per sample) will be taken separately from the surface layer in area C (the peripheral and central parts of the segments) and from the immediate vicinities of the heater (area B) and the metal samples (area A) so as to allow for the evaluation of the spatial stratification of the microbial communities, employing a similar approach to that adopted in the MaCoTe project (Dobrev et al. 2021). The sample post-processing procedure is described in chapter 4.15.2.

4.15   Plan for the analysis of the test materials

4.15.1   Analysis of the metal samples

All the samples will be removed in an anaerobic box, where they will be washed with alcohol and dried to prevent the oxidation of corrosion products to form higher oxidation states during transport. Of the 12 exposed samples from each of the bentonite/exposure time combinations, 10 will be used for determination corrosion weight loss after removal of corrosion product by pickling and 2 will be analysed for corrosion products determination: on the surface, following scraping and in the form of cross-sections.

The samples intended for the determination of the corrosion rate by weight loss will be treated according to the ČSN EN ISO 8407 standard. The steel samples will be immersed in a mixture of hydrochloric acid with water with the addition of a hexamethylenetetramine inhibitor (3.5 g of hexamethylenetetramine (HMTA) solution in 500 ml of 35% concentrated hydrochloric acid and made up to 1000 ml with distilled water) and the copper samples in a sulphuric acid solution (100 ml of sulphuric acid and 900 ml of distilled water). Each sample will be subjected to a minimum of five treatment steps, or until constant weight losses have been attained over time. The samples will then be used to determine the localisation of uniform corrosion. Two samples will be analysed via optical profilometry to determine the areal distribution of the

localised penetrationand will then be embedded in acrylate resin and observed in the form of a perpendicular sections so as to determine the maximum depth of the penetration. In case of severe localized corrosion, another two samples will be involved in the optical profilometry in order to collect reliable data in terms of statistics.

The two samples intended for the analysis of corrosion products will not be subjected to the above treatment procedure. The layer of corrosion products on the surface will first be analysed by means of X-ray diffraction (XRD) followed by Raman spectroscopy so as to determine the phase composition of the corrosion products. The corrosion products will be scraped from a section of the surface, while the corrosion products further from the surface will be analysed using X-ray diffraction. Subsequently, both samples will be embedded into epoxy resin in an evacuated desiccator in the direction perpendicular to the surface of the sample base. The epoxy resin will allow for improved flow conditions and the subsequent consolidation of the corrosion products for grinding. The samples will be ground to a fineness level of P4000 sand paper. Diamond paste polishing will not be applied so as to avoid the effect of the presence of the paste in the pores of the corrosion products on the results of the analysis. Similarly, the samples will not be etched so as to prevent the etching of minor components of the corrosion products. The elemental composition will be monitored on a vertical section via the SEM/EDS method, possibly supplemented by local phase analysis applying the X-ray microdiffraction (µXRD) method.

4.15.2 Analysis of the bentonite

From the point of view of the bentonite investigation in the planned in-situ experiments, it is desirable to analyse:

area A: bentonite in the immediate vicinity of the metal samples,

area B: bentonite in the immediate vicinity of the heater,

area C: bentonite in the central and peripheral parts of the bentonite segments,

and compare with the bentonite input material parameters (see chapter 4.3.2).

The planned analyses are the same as that for the initial characterisation of the bentonite materials (see chapter 4.3.2), i.e. X-ray fluorescence spectroscopy (XRF), the determination of the carbon and sulphur species (TOC, TIC and TS), thermal analysis (TA-EGA), powder X-ray diffraction (XRD) analysis, the determination of the specific surface area (SSA), the swell index (SI) and the cation exchange capacity (CEC) and the analysis of leachable ions. As mentioned in chapter 4.3.2, due to the monitoring of the reproducibility of the results and thanks to the simple execution, the CEC, SSA and SI determinations will be performed on a set with real samples taken from the in-situ experiments. Therefore, this section provides a description of the SSA and SI determination procedures. Furthermore, the possibility of performing the analysis of leachable ions and the CEC determination under anaerobic conditions and the resulting benefits are outlined.

- Specific surface area (SSA)

The specific surface area of powdered materials is determined via gas absorption (usually nitrogen). Since no gases can be absorbed by bentonite materials (gas molecules are unable to access the montmorillonite interlayer), a procedure that uses a polar liquid (ethylene glycol-monoethyl ether (EGME)), which, in the form of a molecule monolayer, is able to access the

montmorillonite interlayer will be applied. Powdered samples will be equilibrated in a desiccator with a $CaCl_2$-EGME solvate followed by the removal of the excess EGME via evacuation. The surface of the samples will be covered with a monolayer of EGME molecules once equilibrium is reached between the EGME vapour and the EGME adsorbed on the samples. The amount of EGME adsorbed and thus the specific surface area will then be determined from the change in the weights of the samples. The determination procedure is described in detail in, for example, Brázda and Červinka (2016). Our experience with this determination approach indicates that it is advisable to include a sample with a known specific surface area in the sample set. Therefore, this parameter will be determined in a similar way to that of the CEC, i.e. via a set of both real samples from the in-situ experiments and the input bentonite samples. Thus, it will be possible to monitor the reproducibility of the SSA determination via the results obtained for the input material samples and to draw reliable conclusions on changes in the specific surface area.

- Swell index (SI)

The determination of the swell index provides a useful indicator of the swelling and erosion properties of bentonite materials. The determination of the SI is simple and serves to clearly illustrate changes in the swelling and erosion properties of the material via changes in the exchangeable cation or montmorillonite contents. Therefore, this parameter will be determined for a set of real samples taken from the in-situ experiments with an input sample of bentonite as the reference. Bentonite is left in contact in a graduated cylinder filled with distilled water of a selected volume (most often 2 g and 100 mL). The volume of the swollen bentonite is then read from the scale on the measuring cylinder for a selected time interval. This test also provides information on the erosion behaviour of the bentonite. The presence of colloidal particles that lead to bentonite erosion is also recorded.

- Analysis of leachable ions

Real samples of bentonite from areas C and B will be subjected to the analysis of leachable ions. The principle and procedure of this method is described in chapter 4.3.2. The only modification concerns the potential to perform the procedure even under anaerobic conditions according to the required objective. The determination of this parameter under aerobic conditions will allow for a comparison with the results of the initial characterisation of the bentonites, especially the change in the content of leachable ions during the conducting of the in-situ experiment. The aerobic determination is more accurate (in the sense of the control of the phase ratio) and easier to perform than under anaerobic conditions, where it is necessary to take into account a lower degree of accuracy (the weight is determined here according to the digital pre-weights); on the other hand, no shift is evident in the equilibrium of the carbonate phases (the absence of CO_2 in the anaerobic box compared to aerobic conditions) or changes in the iron oxidation state. Therefore, in order to obtain the maximum amount of information, a combination of the two approaches will be applied.

- CEC

Bentonite samples from all the areas of the experiment (A, B and C) will be subjected to the determination of the cation exchange capacity (for principle and determination procedure see chapter 4.3.2). The selected approach allows for the analysis of all the samples at once and in one set that includes an input material sample. It will be possible to monitor the reproducibility of the determination method on samples of the input materials, thus allowing for the drawing

of reliable conclusions on changes in the CEC. As with the analysis of leachable ions, the determination procedure can be modified to the anaerobic environments. The displacement of exchangeable cations in the anaerobic environment and the use of methods for distinguishing the oxidation state of iron will allow for the monitoring of the fate of iron in the experiment, especially in area A. However, it is important to note that this procedure is not yet used on a routine basis, albeit, in principle, not being difficult to qualitatively determine the oxidation state of iron. The quantitative determination of the Fe^{2+} and Fe^{3+} concentrations would require the optimisation of the method and the validation of the determination technique.

- Microbiological analysis

The processing of compacted bentonite samples (cutting into pieces, slicing, etc.) for the microbiological analysis to be conducted at the Technical University of Liberec Institute for Nanomaterials, Advanced Technology and Innovation will take place under sterile conditions; the samples will be handled in anaerobic glove boxes, which will be thoroughly sterilised beforehand by the wiping of all the surfaces (detergent and ethanol) and the use of a germicidal lamp. All the instruments that come into contact with the bentonite samples will be heat sterilised. The next step prior to the analysis will comprise the isolation of the DNA from the bentonite samples, which will require the homogenisation of the bentonite under sterile conditions. For DNA isolation, bentonite samples will be processed in accordance with the study of Engel et al. (2019) so that the results from PKE will be comparable to the results of the MaCoTe project. The DNA from the bentonite samples will then be isolated using a kit designed for the isolation of nucleic acids from soil (DNeasy PowerMax® Soil DNA Isolation Kit (Qiagen)); the isolation process will follow the manufacturer's instructions. The microbial diversity in the bentonite samples will be studied using DNA molecular genetic methods. The concentration of the isolated DNA will then be measured fluorometrically using a Qubit® 2.0 Fluorometer (Invitrogen, Life technologies, USA), again according to the manufacturer's instructions. However, it will be necessary to take into account the expected extremely low DNA yields from the bentonite samples, i.e. the concentration values of the isolated DNA will, in most cases, be below the measurement detection limit. Hence, the quantification of copies of the 16S rRNA gene by means of the qPCR and the determination of the Cq value will be used as a measure of the presence of amplifiable DNA (the amplification cycle wherein the amplification and fluorescence detection of DNA copies during the PCR occurs to a degree beyond the background level) in the samples. If the Cq value is similar to that of the no-template control (NTC), the isolation of the DNA will be considered unsuccessful and the sample will not be further genetically analysed. The universal 16S rRNA primers 16SqPCR-F and 16SqPCR-R will be used for evaluation purposes (Nadkarni et al. 2002).

The samples that pass the qPCR DNA isolation quality control test described above will be further sequenced applying the amplicon sequencing of the 16S rRNA gene. This method allows for the detailed determination of the qualitative composition and structure of the whole of the community and has been described in detail in an interim technical report compiled by Černík et al. (2015) and in various publications (see Černoušek et al. 2020; Shrestha et al. 2022). The IonTorrent platform (Life Technologies, USA) will be employed. The specific molecular marker will then comprise the variable V4 region of the 16S rRNA gene using 530FA primers (Dowd et al. 2008) and 802R (Claesson et al. 2009).

QIIME 2 software version 2021.8 (Bolyen et al. 2019) will be used for the processing of the raw microbiological molecular genetic NGS data (sequences). Low quality, chimeric and overly

short sequences that have not been read by the end of the required section will be removed from the NGS data set via quality filtering using the q2-demux and q2-dada2 plug-in modules (Callahan et al. 2016). The final sequences will be free of unwanted noise. Unique sequence variants (so-called ASVs - amplicon sequence variants) will be created from the resulting purified sequences via the q2-feature-classifier plug-in (Bokulich et al. 2018) using the "classify-sklearn naive Bayes" algorithm against the SILVA database version 138 (Quast et al. 2012). ASVs are gradually replacing the previously standard OTUs in expert studies (Callahan et al. 2016). The sequences that occur only once in the dataset will not be further evaluated since they will most likely comprise artifacts (sequencing errors) of the method. Finally, representative sequences (ASV) will be compared with a publicly-available database - Genbank and Silva (taxonomic classification) or the available literature (the metabolic characterisation of microorganisms). The QIIME 2 outputs will be processed in the R using the Phyloseq package (McMurdie and Holmes 2013). Classic one-dimensional (column, boxplots, etc.), recently introduced "bubble" (FAPROTAX) and ordination (multidimensional) diagrams will be used for the final visualisation of the results of the analysis.

4.15.3   Analysis of the groundwater

- Determination of the chemical composition of the groundwater

The cations in the groundwater will be determined via the AAS and ICP-MS methods according to the ČSN EN ISO 17294-2 standard. The anions will be determined according to the ČSN EN ISO 10304-1 standard using the ion chromatography method and the determination of the total alkalinity by means of potentiometric titration according to the ČSN EN ISO 9963-1 and ČSN 75 7373 standards.

- Determination of the electrochemical parameters of the groundwater

The measurement of the pH, redox potential, conductivity and residual oxygen electrochemical parameters will be performed directly at the Bukov URF using a special flow cell (Fig. 8) with a WTW Multi 3630 IDS device (WTW, Germany). The electrodes will be calibrated prior to measurement and replaced with new ones as required during experimentation.

The electrochemical parameter measurement cell will be flushed with argon, following which, groundwater will be forced into the cell using argon through the sampling device and filtered. The relevant parameters will subsequently be measured under "anaerobic conditions" (flushing with argon will not completely remove the oxygen from the system).

- Determination of the colloidal particles in the groundwater

The colloidal particles will be determined under laboratory conditions using a Nanophox device (Sympatec GmbH, Germany). Due to changes in the temperature and transport of the groundwater, the measurement of colloids will focus only on confirming their presence in the system since the particle size distribution curve may be skewed as a result of coagulation due to the conditions that prevail in the boreholes during the experiment.
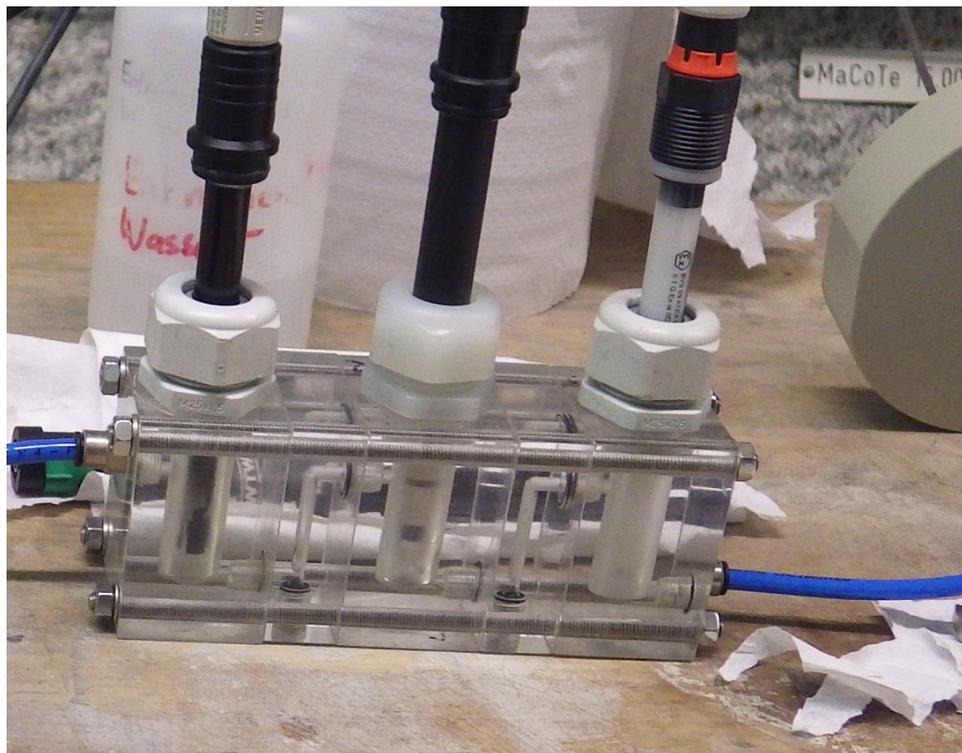
Fig. 8 Illustration of the electrochemical cell

- Microbiological analysis of the groundwater

The procedure for the processing of the groundwater samples for the microbiological molecular genetic analysis will be very similar to that of the bentonite analysis, see chapter 4.15.2. The first step of the analysis will comprise the isolation (extraction) of DNA from the groundwater samples via filtering and capture on Sterivex filters; the filters will be removed from the plastic capsule using a sterile pipe cutter. The PowerWater DNA Isolation Kit (Qiagen/MoBio, USA) will be used for the isolation of the DNA, and the isolation process will follow the manufacturer's instructions. The concentration of the isolated DNA will then be measured fluorometrically using a Qubit® 2.0 Fluorometer (Invitrogen, Life technologies, USA), again according to the manufacturer's instructions. The isolated DNA will be used for amplicon sequencing (according to the procedure described in chapter 4.15.2) and the quantification of specific groups of microorganisms by means of the qPCR.

NGS analysis will be employed mainly with respect to the (relative and absolute) quantification of specific functional groups of microorganisms, sulphate-reducing bacteria (SRB), denitrifying bacteria (e.g., NRB), iron-reducing bacteria (IRB) and the total bacterial biomass, and will supplement the standard quantitative PCR method (qPCR and Real-time PCR). The analysis will be performed using a LightCycler ® 480 device (Roche Biochemicals, USA). One of the disadvantages of qPCR concerns its absolute quantification that requires a standard (the comparison of the Cq values on a standard curve); moreover, the determination of a relevant standard for environmental samples that contain a mixture of various microorganisms of unknown ratios is particularly challenging, if not impossible (Dhanasekaran et al. 2010) – a problem that is no longer being addressed in the literature. It is therefore planned to apply relative quantification, i.e. comparison with the first sampling campaign in the various boreholes, which will serve to determine changes over time rather than the exact number of cells.

Molecular markers specific to the various organisms will be used to determine the occurrence of specific functional groups of microorganisms in the samples, namely the RH1-dsr-F and RH3-dsr-R primers (Ben-Dov et al. 2007) for sulphate-reducing bacteria, gene encoding dissolved N_2O reductase for denitrifying bacteria, namely the nosZ-F and nosZ 1622R primers (Geets et al. 2007) and specific 16S primers that target Geobacteraceae, which include a large number of iron-reducing bacteria (IRB), namely the Geo494F and Geo825R primers (Anderson et. 1998, Holmes et al., 2002). The universal 16S rRNA primers 16SqPCR-F and 16SqPCR-R will be used to evaluate the total bacterial biomass (Nadkarni et al. 2002). Subsequent bioinformatics analyses will be performed in the same way as in the case of bentonite data in chapter 4.15.2.

5 Expected results of the corrosion experiment

5.1 Expected results concerning corrosion

The weight loss results will provide information on the average corrosion rate, while the optical profilometry and cross-sectional evaluation will provide information on the localisation of the corrosion attack (Dobrev et al. 2020). In both cases, time series will be available to allow for the verification and refinement of previously-developed long-term models (Forman et al. 2021) of waste disposal packages. The determination of the localisation of corrosion attack and subsequent developments will allow for the revision of the corrosion addition to the thickness of the wall of the WDP.

Knowledge of the composition of the corrosion products is essential for the accurate setting of long-term models since the main associated event concerns the transport of ions through newly formed layers of corrosion products. The overall volume of the corrosion products formed and their porosity comprise the main parameters that determine the rate of corrosion attack (Dobrev et al. 2020, Forman et al. 2021).

The determination of the occurrence of specific corrosion products (Gondolli et al. 2018) in conjunction with the microbiological analysis helps to determine the effect of microorganisms on the rate and localisation of corrosion (e.g. mackinawite FeS is formed by sulphate-reducing bacteria and the ferric corrosion products goethite and lepidocrocite $FeOOH$, again with the contribution of denitrifying bacteria). The formation of silicate corrosion products is evidence of the hydrolysis of water in a limited volume of electrolyte on the surface of steel and a local decrease in the pH. This phenomenon leads to the localisation of corrosion attack; however, this is a temporary process. It is the formation of silicates (greenalite, cronstedtite, berthierine) that significantly retards localised corrosion attack (Forman et al. 2021).

5.2 Expected results concerning the bentonite

The expected results concerning the bentonite materials are summarised in Tab. 1. Due to the different montmorillonite content and dominant exchangeable cation in the two bentonites, we expect different results with some respects. However, overall, only small changes in the monitored parameters at the metal sample/bentonite and heater/bentonite interfaces can be expected. We also estimate that these small changes at the interfaces will be negligible in terms of the total bentonite mass (area C).

Tab. 1 Estimation of the probability of the occurrence of the expected results for both types of bentonites

Bentonite	BCV	BARA KADE
Bentonite chemical erosion	unlikely	likely the bulk density of the bentonite decreases due to the transport away of eroded bentonite particles

Bentonite	BCV	BARA KADE
Reduction in the bulk density	likely in the case of the technological gaps and the deformability of the apparatus, swelling can be expected and thus a reduction in the bulk density of the bentonite (and the relevant derived parameters – moisture content, porosity, etc.)	likely in the case of the technological gaps and the deformability of the apparatus, swelling can be expected and thus a reduction in the bulk density of the bentonite (and the relevant derived parameters – moisture content, porosity, etc.)
Changes in the chemical composition of bentonite	unlikely very small changes can be expected, which will be around the limit of quantification. In the case of more significant changes in the proportion of exchangeable cations, this will be reflected in the chemical composition. In the case of significant microbial activity, changes in the carbon- and sulphur-containing phases can be expected.	unlikely very small changes can be expected, which will be around the limit of quantification. In the case of more significant changes in the proportion of exchangeable cations, this will be reflected in the chemical composition. In the case of significant microbial activity, changes in the carbon- and sulphur-containing phases can be expected.
Changes in the mineralogical composition	unlikely very small changes can be expected, which will be around the limit of quantification. Mineralogical changes can be expected on the contacts of the bentonite with the heater and the corrosion samples. Under the hydrothermal conditions, the leaching of soluble mineral phases and a slight increase in montmorillonite content (and the relevant derived parameters - CEC,	unlikely very small changes can be expected, which will be around the limit of quantification. Mineralogical changes can be expected on the contacts of the bentonite with the heater and the corrosion samples. Under the hydrothermal conditions, the leaching of soluble mineral phases and a slight increase in montmorillonite content (and the relevant derived parameters - CEC,

Bentonite	BCV	BARA KADE
	SSA_EGME) can be expected.	SSA_EGME) can be expected.
Changes in the leachable ion concentrations	likely under hydrothermal conditions, the leaching of the soluble mineral phases can be expected, thus acting to reduce the amount of leachable ions.	likely under hydrothermal conditions, the leaching of the soluble mineral phases can be expected, thus acting to reduce the amount of leachable ions.
Changes in the representation of exchangeable cations	likely the equilibration of bentonite with the groundwater will lead to cation-exchange reactions that lead to changes in the proportion of exchangeable cations.	likely the equilibration of bentonite with the groundwater will lead to cation-exchange reactions that lead to changes in the proportion of exchangeable cations.

- Microbiology results

The expected result with respect to the bentonite microbiology will comprise the determination of the qualitative composition of the microbial consortia focusing on both the taxonomic structure and the functional structure, concerning which the dominant taxa will be traced to their functional profiles. Emphasis will be placed on microorganisms that have the potential to affect (accelerate) the corrosion of the WDP and, especially, on those microorganisms that could theoretically affect the structure of the bentonite (e.g. IRB). Thanks to the use of the qPCR method, it will be possible to monitor changes in the abundance of all the microorganisms over time as well as changes in the various functional groups over time. The amount of microbial DNA and the Cq values of the 16S rDNA gene obtained from qPCR analysis will provide indicators of the amount of the microbial biomass.

5.3 Expected results concerning the groundwater

With respect to the electrochemical parameters, we assume that the pH will be above 7 due to the dissolution of carbonates from the bentonite, which will act to enhance the conductivity of the groundwater (Dobrev et al. 2021). The oxidation-reduction potential, following conversion to a standard hydrogen electrode, should evince negative values, and the residual oxygen should be on the limit of detection. In the long term, there should be a decrease in the conductivity due to a reduction in the concentration of dissolved carbonates. The long-term development of the pH will largely depend on the metabolic products of the microorganisms, e.g., the pH could fall to slightly below 7 due to the production of acetates.

Concerning the chemical analysis of the groundwater, we mainly expect an increase in the concentration of sodium and bicarbonates due to their dissolution from the bentonite. Their concentration is then likely to decline over the long term.

We assume that there will be more colloidal particles in the BARA KADE groundwater than in that of the Czech BCV bentonite.

The trends identified in the behaviour of the groundwater, combined with the results of the bentonite analysis, will assist in the prediction of the long-term behaviour of the bentonite.

The expected result with respect to the groundwater microbiology will concern the determination of the qualitative composition of the microbial consortia focusing on both the taxonomic structure and the functional structure, concerning which the dominant taxa will be traced to their functional profiles. Emphasis will be placed on microorganisms that have the potential to affect (accelerate) the corrosion of the WDP. Thanks to the use of the qPCR method, it will be possible to monitor changes in the abundance of all the microorganisms over time as well as changes in the various functional groups over time. The amount of microbial DNA and the Cq values of the 16S rDNA gene obtained from qPCR analysis will provide indicators of the amount of the microbial biomass.

6 Uncertainties of the experiment

6.1 Uncertainties concerning corrosion

The uncertainties involved in determining the rate of corrosion attack are of two types. Firstly, factors that influence the overall corrosion attack. This category includes, in particular, the transport of dissolved corrosion products, which is mainly affected by the level of compaction of the bentonite and the phase composition of the corrosion products. Secondly, the localisation of the corrosion attack is influenced by a wide range of factors, the most important of which comprise uneven bentonite saturation by groundwater, the presence of residual oxygen, the inhomogeneous microstructure of the steel, the presence of preferential pathways in the bentonite and the action of microorganisms.

The use of very pure steel without carbide-forming elements would significantly reduce the influence of the microstructure. The employed experimental setup would essentially eliminate the effect of residual oxygen and uneven saturation since the cells would be both deaerated and saturated with demineralised water under anaerobic conditions.

1) Local bulk density of the bentonite

The planned final bulk density following the swelling of the bentonite is required to correspond to 1600 kg m^{-3}. However, this concerns the average bulk density; it is possible that the bulk density in the vicinity of the technological gaps (of around 0.1 mm) will be lower locally. However, this factor corresponds to real conditions, i.e. there will also be a technological gap between the WDP and the bentonite following disposal in the DGR (Forman et al. 2021). In addition, lower local bulk density values can be considered somewhat conservative since the corrosion rate decreases with increasing bulk density over time (Forman et al. 2021; Smart et al. 2017; Marsh and Taylor 1988).

2) Composition of the corrosion products

The phase composition of the corrosion products is a crucial factor in the determination of the lifetime of the WDP. While short-term models can be successfully verified armed with knowledge on the composition of short-term metastable corrosion products, it is possible that a ten-year exposure period is too short for attaining knowledge on thermodynamically stable corrosion products. However, as indicated in the previous paragraph, the use of models based on short-term data represents a conservative approach since long-term stable corrosion products have a larger volume and act to reduce both transport and the overall corrosion rate.

In addition, a possibility of corrosion products layer cracking during long-term exposure has to be taken into the consideration as well.

3) The "scale effect" and the absence of preferential pathways

The small dimensions of the experiment act to practically eliminate the potential for uneven saturation and the creation of preferential paths (cracks in the bentonite), which could arise in larger-dimension experiments. This factor has two aspects. On the one hand, the conditions of the experiment provide for a sealed system that corresponds to the state of the disposal system in later disposal periods, while on the other, it results in incomplete data on the effect of the localisation of corrosion attack at the beginning of the disposal period.

4)　Microbial corrosion

The localisation of corrosion can be affected by microorganisms. Although a detailed microbiological analysis of the bentonite and (during the course of the experiment) the analysis of the surrounding water will be available after the defined time periods, it should be borne in mind that the development of microbial consortia is a very dynamic phenomenon. Even slight differences in the conditions of the boreholes may lead to the development of different consortia. Significant corrosion attacks may thus be initiated by bacterial strains that may only act over a short time period in this experiment. In terms of corrosion attack, 5 time points over 10 years is insufficient to determine the kinetics of localised attack caused by microorganisms.

This aspect comprises a key factor. Although the experiment is designed to allow for the thorough analysis and description of microbial processes, this uncertainty needs to be taken into account. The solution would be to apply a technique that allows for the continuous monitoring of the corrosion attack, e.g. resistometry or electrochemical linear polarisation resistance; however, such methods are technically very difficult to apply in the considered experimental setup since they require electrical contacts outside the boreholes; moreover, such contacts are notoriously prone to mechanical failure over the long term.

6.2　Uncertainties concerning the bentonite

Obtaining a sufficient number of samples to perform all the planned analysis appears to be the main uncertainty in terms of the post-experimental bentonite analysis phase. This uncertainty is most significant when taking bentonite from around the metal samples. It should also be noted that since priority will be accorded to obtaining sufficient bentonite sample volumes regardless of the position of the bentonite in the modules (areas A, B and C), it will probably not be possible to verify the occurrence of potential spatial inhomogeneities.

The determination of the moisture content of the bentonite following the end of the experiment will be burdened by the uncertainty caused by the immediate spontaneous drying of the samples in the glove box. Therefore, the determined values will be lower than the actual moisture content state in the experiment.

It may not be possible to detect the mineralogical changes in the randomly-oriented bentonite samples via XRD due to not reaching the detection limit or the formation of amorphous phases (e.g. Svensson et al. (2011, 2017)). The determination of leachable ions and the CEC and SSA requires relatively large samples, and it is possible that there will not be a sufficient amount of bentonite for the whole of the planned analysis. Moreover, in general, one of the potential problems of the planned analysis methodologies is that their quantification limits will be higher than the changes in the bentonite, thus rendering them unquantifiable.

When determining the CEC in the area of the metal samples, the results may be skewed due to the "contamination" of the samples from these areas by the surrounding bentonite or corrosion products.

With respect to certain parts of the analysis, it may not be possible to interpret the results with clear justification for the reasons for the changes that have occurred, i.e. whether the changes are due to transport processes, chemical reactions or microbial activity in the system. Changes in the TOC provide such an example.

As in the case of the groundwater (see below), the ubiquitous risk of contamination is also relevant for bentonites from the microbiological point of view (bentonites comprise extremely poor environments in terms of biomass). In addition, the molecular genetic analysis (*inter alia*) will be blocked by the ability of bentonite to bind various reagents to its DNA surface. Finally, the results of the bentonite sequencing analysis will be significantly more difficult to interpret than those of the groundwater samples due to the extremely low yields of DNA that will be obtained from the bentonite samples. The DNA concentration values are expected to be below the measurement detection limit. Low concentrations of input DNA generally lead to increased proportions of contamination. Therefore, in addition to the direct isolation of DNA from bentonite powder (with the significant risk of not acquiring enough quality DNA from the material), the bentonite will be screened in the suspended state, allowing time for the development of microorganisms and the subsequently higher chance of their capture and identification.

6.3 Uncertainties concerning the groundwater

Any uncertainties in the results of the chemical composition and electrochemical parameters of the groundwater will be due to differences in the conditions in the boreholes. The identification of significant differences between the boreholes will complicate the comparison of the long-term development of the various parameters.

The measurement of the redox potential may be affected by the residual oxygen in the cell following flushing with argon. Due to the fact that exclusively anaerobic conditions cannot be technically guaranteed, the determined values may differ to those determined for the boreholes.

Analytical methods generally have quantification limits; if the values lie below these limits, it will not be possible to quantitatively interpret the results. However, the presence of a given substance is not excluded if it is below this limit, i.e., in the case of values below the limit of detection it will not be possible to determine whether the analyte is present in the water or not unless the laboratory analysis provides us with more detailed information.

Concerning the measurement of colloidal particles, any uncertainties will be associated with changes in the conditions in the boreholes and the time lag between the sampling and the analysis. Furthermore, the evaluation of colloidal particles is based on Rayleigh scattering, i.e. for spherical particles, whereas colloidal bentonite particles should not have a spherical shape.

A number of uncertainties also relate to the collection and evaluation of the microbiological groundwater samples. With respect to the monitoring of the groundwater, due to the insufficient time allowance between the drilling of the 11 experimental boreholes and the requirement of the contract to submit results for the first year of experimentation, the first-year results will be based on the overly-frequent extraction of samples compared to the less frequent sampling of the groundwater for the longer-term experimental modules. As in the case of the bentonites, the ubiquitous risk of contamination is relevant to the groundwater (also due to the usually very poor biomass environment of the samples). In the event that larger colloids are present in the water, the volume of water collected and actually filtered may fall below the optimal 300-1000 ml.

6.4 Uncertainties concerning the technical design and the location of the boreholes

It will be possible only to minimise (rather than eliminate) the uncertainties inherent in the technical design of the experiment. This applies particularly to the uniformity of the temperature through the whole of the vertical profiles of the modules. It is probable that due to variations in the dissipation of heat in the horizontal and vertical directions, the temperatures of the various levels and the samples will not be identical. However, it is expected that such differences and the effect on the uniformity of the temperature will not be significant. Although the experimental assembly will be tested under laboratory conditions, the actual temperature differences will only be known after the assemblies have been emplaced in the boreholes.

The groundwater sampling device is designed so that water is gradually forced out of the area beneath the mechanical obturator towards the modules. It is likely that the water temperature beneath the obturator will differ to that of the water in the vicinity of the modules; moreover, it is possible that the microbial communities will be stratified according to the depth of the boreholes. In addition, the extraction process is likely to lead to the extraction of mixed samples of groundwater and, thus, the results of the analysis will reflect the "average" composition rather than the stratified composition in the borehole.

Due to differences in the local fracture systems, it is likely that different conditions will pertain in the various boreholes, which may affect the microbial communities, thus affecting the accuracy of the comparison of the long-term development in the boreholes.

Should the extraction of groundwater affect the conditions in the boreholes (the inflow of oxygenated water, etc.), we will be faced with considerable uncertainty as to whether the system would have developed in the same way from the microbiological point of view as it would had the system not been disturbed.

7 References

ANDERSON R. T., ROONEY-VARGA J., GAW C. V., LOVLEY. D. R. (1998): Anaerobic benzene oxidation in the Fe(III)-reduction zone of petroleum-contaminated aquifers. – Environ. Sci. Technol. 32, 1222–1229.

BEN-DOV E., BRENNER A., KUSHMARO A. (2007): Quantification of sulfate-reducing bacteria in industrial wastewater, by real-time polymerase chain reaction (PCR) Using dsrA and apsA Genes. – Microbial Ecology 54, 439–451.

BOKULICH N. A., KAEHLER B. D., RIDEOUT J. R., DILLON M., BOLYEN E., KNIGHT R., CAPORASO J. G. (2018): Optimizing taxonomic classification of marker-gene amplicon sequences with QIIME 2's q2-feature-classifier plugin. – Microbiome, 6, 1–17.

BOLYEN E., RIDEOUT J. R., DILLON M. R., BOKULICH N. A., ABNET C. C., AL-GHALITH G. A., CAPORASO J. G. (2019): Reproducible, interactive, scalable and extensible microbiome data science using QIIME 2. Nature biotechnology, 37(8), 852−857.

BRAZELTON W.J., NELSON B., SCHRENK M.O. (2012): Metagenomic Evidence for H2 Oxidation and H2 Production by Serpentinite-Hosted Subsurface Microbial Communities. Front. Microbiol. 0. https://doi.org/10.3389/fmicb.2011.00268

BRÁZDA L., ČERVINKA R. (2016): Determination of specific surface area of clay minerals by EGME method. Proceedings of the 4th BELBaR Annual Workshop, Berlin, Germany, 15 September 2016.

CALLAHAN B. J., MCMURDIE P. J., ROSEN M. J., HAN A. W., JOHNSON A. J. A., HOLMES S. P. (2016): DADA2: High-resolution sample inference from Illumina amplicon data. – Nature Methods 13, 581−583.

CASAR C.P., KRUGER B.R., OSBURN M.R. (2021): Rock-Hosted Subsurface Biofilms: Mineral Selectivity Drives Hotspots for Intraterrestrial Life. Front. Microbiol. 0. https://doi.org/10.3389/fmicb.2021.658988

CLAESSON M. J., O'SULLIVAN O., WANG Q., NIKKILA J., MARCHESI J. R., SMIDT H., DE VOS W. M., ROSS R. P., O'TOOLE P. W. (2009): Comparative analysis of pyrosequencing and a phylogenetic microarray for exploring microbial community structures in the human distal intestine. – PLoS One.

ČERNÍK M., SHRESTHA R, STEINOVÁ J., STOULIL J., ŠEVCŮ A., ŠPAČEK P. (2015): Mikrobiální koroze za podmínek hlubinného úložiště pro koncepci ocelový UOS – zhutněný bentonit – Rešeršní práce a plán experimentů – Průběžná technická zpráva. Zpráva SÚRAO, 31/2015, 73 str.

ČERNOUŠEK T., SHRESTHA R., KOVÁŘOVÁ H., ŠPÁNEK R., ŠEVCŮ A., SIHELSKÁ K., STEINOVÁ J. (2020): Microbially influenced corrosion of carbon steel in the presence of anaerobic sulphate-reducing bacteria. Corrosion Engineering, Science and Technology, 55(2), 127−137.

ČERVINKA R. (2014): Stanovení kationtové výměnné kapacity (CEC) a výměnných kationtů pomocí metody s Cu(II)triethylentetraminem. Certifikovaná metodika. Č.j. SÚJB/ONRV/15452/2014.

ČERVINKA R., VEČERNÍK P., KAŠPAR V., VAŠÍČEK R. (2018): Kompletní charakterizace bentonitu BCV 2017 - průběžná zpráva. SÚRAO Technická zpráva 419/2019, Česká republika, 33 s.

DEBRUYN W., DRESSELAERS J., VERMEIREN PH., KELCHTERMANS G., TAS H. (1991): Corrosion of container and infrastructure materials under clay repository conditions. Final report to the CEC for the period 1985–1989. Contract FII W/0033-B, EUR13667EN. Mol, Belgium.

DHANASEKARAN S., DOHERTY T.M., KENNETH J., TB TRIALS STUDY GROUP (2010): Comparison of different standards for real-time PCR-based absolute quantification. J. Immunol. Methods 354, 34–39. https://doi.org/10.1016/j.jim.2010.01.004

DOBREV D., BÁLEK V., BRŮHA P., ČLUPEK M., KISELOVÁ M., KOUŘIL M., NOVÁK P, SILBER R., STOULIL J., VEČERNÍK P. (2011): Výzkum a vývoj metodik hodnocení degradace materiálu obalových souboru, Zpráva ÚJV 13796, 49 str.

DOBREV D., GONDOLLI J., KOUŘIL M., MENDOZA MIRANDA A.N., STOULIL J. STRAKA M. (2020): Korozní zkoušky s materiály navrženými pro konstrukci ukládacích obalových souborů, SÚRAO TZ 248/2018 rev. č. 1, 956 str., Husinec - Řež

DOBREV D., MENDOZA MIRANDA A. N., STEINOVÁ J., ZUNA M. (2021): Korozní zkouška v přírodním granitoidním prostředí, SÚRAO TZ 194/2017 rev. č. 4, 466 s, Praha.

DOWD S. E., CALLAWAY T. R., WOLCOTT R. D., SUN Y., MCKEEHAN T., HAGEVOORT R. G., EDRINGTON T. S. (2008): Evaluation of the bacterial diversity in the feces of cattle using 16S rDNA bacterial tag-encoded FLX amplicon pyrosequencing (bTEFAP). – BMC Biol., 6.

ENGEL, K., COYOTZI, S., VACHON, M. A., MCKELVIE, J. R., & NEUFELD, J. D. (2019): Validating DNA extraction protocols for bentonite clay. – Msphere, 4(5), e00334-19.

FORMAN L., PICEK M., DOBREV D., GONDOLLI J., MENDOZA MIRANDA A.N., STRAKA M., KOUŘIL M., STOULIL J., MATAL O., ČERMÁK J., KRÁL L., ŽALOUDEK J., VÁVRA M, ČUPR M. (2021): FINAL TECHNICAL REPORT ON THE RESEARCH PART OF THE PROJECT: Research and development of a disposal package for the deep disposal of spent nuclear fuel to the sample implementation stage, Prague, ZZ 544/2021

GEETS J., DE COOMAN M., WITTEBOLLE L., HEYLEN K., VANPARYSUS B., DE VOS P., VERSTAETE W, BOON N. (2007): Real-time PCR assay for the simultaneous quantification of nitrifying and denitrifying bacteria in activated sludge. – Applied Microbiology and Biotechnology 75(1), 211–221.

GONDOLLI J., KLAJMON M. KOUŘIL M. (2018): Korozní produkty – Závěrečná zpráva, Zpráva SÚRAO TZ 329/2018, 123 str., Praha

HOFMANOVÁ E., ČERVINKA R., VOPÁLKA D., BABOROVÁ L., BRÁZDA L., PECKOVÁ A., VETEŠNÍK A., VIGLAŠOVÁ E., VAŠÍČEK R. (2019): Transport radionuklidů z úložiště / vstupní parametry a procesní modely pro hodnocení transportu radionuklidů přes inženýrské bariéry: Závěrečná zpráva. – MS SÚRAO, ZZ 420/2019, Praha.

HOLMES D. E., FINNERAN K. T., O'NEIL R. A., LOVLEY D. R. (2002): Enrichment of members of the family Geobacteraceae associated with stimulation of dissimilatory metal reduction in Uranium-contaminated aquifer sediments. – Applied and Environmental Microbiology 68(5), 2300–2306.

KARNLAND O. (2010): Chemical and mineralogical characterization of the bentonite buffer for the acceptance control procedure in a KBS-3 repository. SKB report TR-10-60, Švédsko, 29 s.

MARSH, G. P., TAYLOR, K. J. (1988): An Assesment of Carbon Steel Containers for Radioactive Waste Disposal. Corrosion Science, 28, 289-320.

MCMURDIE P. J., HOLMES S. (2013): phyloseq: an R package for reproducible interactive analysis and graphics of microbiome census data. PloS one, 8(4), e61217.

MIETTINEN H., BOMBERG M., VIKMAN M. (2015): Microbial activation due to addition of electron donors/acceptors in deep groundwaters (No. DELIVERABLE D2.5). MIND.

NADKARNI M. A., MARTIN F. E., JACQUES N. A., HUNTER N. (2002): Determination of bacterial load by real-time PCR using a broad-range (universal) probe and primers set. – Microbiology 148, 257–266.

NYYSSÖNEN M., HULTMAN J., AHONEN,L., KUKKONEN I., PAULIN L., LAINE P., ITÄVAARA M., AUVINEN P. (2014): Taxonomically and functionally diverse microbial communities in deep crystalline rocks of the Fennoscandian shield. ISME J. 8, 126–138. https://doi.org/10.1038/ismej.2013.125

QUAST C., PRUESSE E., YILMAZ P., GERKEN J., SCHWEER T., YARZA P., GLÖCKNER F. O. (2012): The SILVA ribosomal RNA gene database project: improved data processing and web-based tools. – Nucleic acids research, 41(D1), D590-D596.

ROBADOR A., MÜLLER A.L., SAWICKA J.E., BERRY D., HUBERT C.R., LOY A., JØRGENSEN B.B., BRÜCHERT, V. (2016): Activity and community structures of sulfate-reducing microorganisms in polar, temperate and tropical marine sediments. ISME J. 10, 796–809.

SHRESTHA R., CERNA K., SPANEK R., BARTAK D., CERNOUSEK T., SEVCU A. (2022): The effect of low-pH concrete on microbial community development in bentonite suspensions as a model for microbial activity prediction in future nuclear waste repository. Science of The Total Environment, 808, 151861.

SMART N. R., REDDY B., RANCE A. P., NIXON D. J., FRUTSCHI M., BERNIER-LATMANI R., DIOMIDIS N. (2017): The anaerobic corrosion of carbon steel in compacted bentonite exposed to natural Opalinus Clay porewater containing native microbial populations. Corrosion Engineering, Science and Technology, 52, 101-112.

SVENSSON D., DUECK A., NILSSON U., SANDÉN T., LYDMARK S., JÄGERWALL S., PEDERSEN K., HANSEN S. (2011): Alternative buffer material. Status of the ongoing laboratory investigation of refence materials and test package 1. SKB report TR-11-06, Švédsko, 146 s.

SVENSSON D., LUNDGREN C., JOHANESSON L.-E., NORRFORS K. (2017): Developing strategies for aquisition and control of bentonite for a high level radioactive waste repository. SKB report TR-16-14, Švédsko, 98 s.

SVENSSON D., ERIKSSON P., JOHANNESSON L-E., LUNDGREN CH., BLADSTRÖM T. (2019): Development and testing of methods suitable for quality control of bentonite as KBS-3 buffer and backfill. SKB report TR-19-25, Švédsko, 165 s.

WU X., HOLMFELDT K., HUBALEK V., LUNDIN D., ÅSTRÖM M., BERTILSSON S., DOPSON M. (2016): Microbial metagenomes from three aquifers in the Fennoscandian shield terrestrial deep

biosphere reveal metabolic partitioning among populations. ISME J. 10, 1192–1203. https://doi.org/10.1038/ismej.2015.185

8 Electronic attachments

Appendix 1 The materials sheet for BCV BENTONITE

Appendix 2 The materials sheet for BARA KADE BENTONITE